OPTIMIZATION AND KINETICS OF LAVENDER ESSENTIAL OIL EXTRACTION: EFFECTS OF FLOWER PRETREATMENT, SALT SOLUTIONS, AND HYDROLAT REUSE

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This study aimed to optimize lavender flower pretreatment, assess the effects of different salt concentrations, and evaluate the use of hydrolat from previous hydrodistillation on essential oil yield and composition. Whole flowers produced as much oil as wet-ground or freshly ground flowers. Contrary to previous studies, NaCl and KCl solutions (3-10%) did not affect maximum oil yield but did alter hydrodistillation kinetics, reducing washing rates and enhancing diffusion by disrupting oil glands and raising the boiling point. Using a mix of recycled hydrolat and fresh water increased oil yield and improved kinetics by enhancing internal oil extraction. The process was successfully modeled using distinct washing and diffusion mechanisms. Both pretreatments of lavender flowers have a significant impact on the essential oil composition. Salt pretreatment enhances the extraction of oxygenated monoterpenes compounds and monoterpenes, while hydrolat addition negatively affects the solubility and volatility of oxygenated monoterpenes. In conclusion, soaking whole lavender flowers in hydrolat-water mixture is an efficient method, reducing both energy and water usage, and lowering operational costs, while simultaneously requiring attention to its impact on the composition.

Keywords: *Lavandula angustifolia*, lavender essential oil, hydrodistillation, hydrolat, distillation kinetics.

Introduction

The *Lavandula genus*, part of the *Lamiaceae* family, includes 29 species [1], primarily grown in mountainous regions around the western Mediterranean, Atlantic islands, North Africa, and Southwest Asia. These species are extensively cultivated globally, including Serbia, for their decorative appeal and aromatic qualities. The most significant species is *Lavandula angustifolia* Mill., formerly *L. officinalis*, commonly known as lavender. This shrubby plant features slender, gray leaves of varying sizes and flowers that range in color from blue to violet [2]. Lavender is valued commercially for its flowers (fresh, dried, or ground) and essential oils, renowned for their medicinal and aromatic properties [3]. Lavender essential oil is valued for its diverse bioactive compounds, known for imparting aromas and therapeutic benefits such as sedative, spasmolytic, antiviral, antidepressant, and antimigraine effects [4,5]. Its unique fragrance and soothing properties make it popular in fragrance, cosmetics, aromatherapy, and food applications.

Approximately 1,500 tons of essential oils are produced annually from various *Lavandula* species and hybrids [6]. Global production of lavender essential oil ranges from 200 to 500 tons per year [4,7]. Lavender essential oil production is influenced by climate, geography, farming practices, and market demand. Major production centers include Bulgaria, France, the UK, and Spain [8], with Bulgaria recently overtaking France as the largest producer, yielding up to 100 tons annually on 3,700 hectares [4]. Other producers like the USA, Russia, and Balkan countries have smaller outputs, but lavender farming is expanding, notably in Serbia [9].

Fresh lavender flowers contain 0.5–6.25% essential oil [10], though some are lost during drying [11]. In dry inflorescences, lavender essential oil content ranges from 0.5% to 9.62% [10]. For *Lavandula* × *intermedia* cv. 'Budrovka' (Serbia, Fruška Gora Mt.), oil content dropped from 1.26% in 2019 to 1.03% in 2021 [9], below the 1.3% minimum set by the European Pharmacopoeia [12]. Lavender yields about 8–30 kg/ha of essential oil, depending on conditions [13]. The global lavender essential oil market grew at a Compound Annual Growth Rate (CAGR) of 5.4% from 2017 to 2022, reaching US$ 109.4 million in 2023, and is projected to rise to US$ 201.6 million by 2033 [14].

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Lavender essential oil is primarily extracted using hydrodistillation and steam distillation, both effective methods for preserving its aromatic and therapeutic qualities. However, the yield and chemical composition of the oil can vary significantly depending on the extraction method [10]. Factors such as the use of hydrolat (known also as hydrosol and floral water), pretreatment method, distillation still size, heating rate, and water-to-flower ratio (hydromodule) also affect the efficiency of the process. For instance, using the hydrolat from a previous hydrodistillation batch combined with fresh water to keep the constant water-to-flower ratio increased the essential oil yield from dry lavender flowers by about 7% due to increasing the hydrophilic oil components dissolved in the hydrolat [15].

Effective preparation is crucial for extracting essential oils from lavender flowers. The most frequently used pretreatment methods are physical and chemical, while biological are rarely used. They help break down essential oil-bearing structures to facilitate the essential oil release. Physical methods include grinding [16], ultrasonication [17], and pulsed electric field [18]. Grinding reduces particle size, exposes oil-bearing structures, and disrupts cell walls. Ultrasonication uses cavitation to enhance solvent access and mass transfer, speeding up oil release. A pulsed electric field causes electroporation of plant cells, promoting oil release and improving mass transfer. Chemical pretreatment uses acids, alkali solutions, salts, or surfactants to enhance extraction efficiency and oil yield. Surfactants improve the solubility of hydrophobic molecules, while salts can enhance oil component recovery by altering the vapor-liquid equilibrium. Yu *et al.* found positive effects with KCl, Na_2SO_4, and $MgSO_4$, while K_2SO_4, NaCl, and $MgCl_2$ had adverse effects on lavender essential oil yield [19]. Filly *et al.* showed NaCl and surfactants like Tween 40 increased lavender essential oil yield and reduced energy consumption compared to the conventional method [16]. Biological methods employ hydrolytic enzymes (e.g., cellulase, pectinase) to break down cell walls and boost essential oil yield [20] on account of higher energy consumption [21].

This study investigates the hydrodistillation of lavender essential oil using a laboratory-scale Clevenger apparatus, focusing on optimizing flower pretreatment and distillation medium modifications to maximize essential oil yield and evaluate process kinetics. The pretreatment methods for lavender flowers include dry and wet grinding and sieving, while the distillation medium modifications involve the addition of salts and surfactants to water, and the use of hydrolat from previous distillation batches mixed with fresh distilled water for soaking the flowers. The primary objectives are to identify the most effective physical pretreatment and distillation medium modification methods.

Experimental

Plant material and chemicals

Dry lavender flowers, harvested in 2022, were purchased from a farm in Starčevo (Serbia). Moisture content, determined by drying in an oven at 105 °C until constant mass, was 7.9%. KCl and NaCl were purchased from Centrohem (Stara Pazova, Serbia), while Tween 40 was obtained from Sigma-Aldrich (Darmstadt, Germany).

Plant material preparation

In this study, whole (unground) flowers, dry ground flowers, sieved (< 1.0 mm), and then stored in a paper bag in a dry, dark place at room temperature until distillation, dry ground flowers and sieved (< 1.0 mm) immediately before distillation, dry ground flowers (not sieved) immediately before distillation, and wet ground flowers were subjected to hydrodistillation.

Flowers were dry-ground using an electric mill (Mahlkoenig, Hamburg, Germany). Approximately 100 g of flowers were processed for about 20 seconds, after which the mill was turned off to prevent overheating and to shake off any powder adhering to the mill walls. The grinding was repeated once more. The resulting flower powder was sieved to obtain a 1.0 mm fraction, while the larger particles were reground using the same mill and sieved again to achieve the same size fraction. The final powder was manually mixed to ensure uniformity.

For wet grinding, a domestic blender (Nutribullet, Model NB-101B, China) was used. A portion of the flowers, corresponding to the distillation flask volume and the water-to-flower ratio, was suspended in approximately half of the required water in the blender jar and blended for 20 seconds. The resulting suspension was then transferred to the appropriate distillation flask. The blender jar was rinsed multiple times with water, and these rinses, containing the remaining flower particles, were also added to the distillation flask, ensuring that the total volume met the desired water-to-flower ratio.

Hydrodistillation

A Clevenger apparatus with a 1000 mL round-bottom flask was used to study the effects of different grinding methods, the addition of salts and surfactants, and the use of a distilled water and hydrolat mixture. Lavender flowers (35 g) were immersed in water at a water-to-flower ratio of 10:1 (mL/g). When the effect of grinding was investigated, hydrodistillation was carried out at an approximate distillation rate of 7 mL/min.

To assess the impact of salts (KCl and NaCl) and Tween 40, aqueous solutions were prepared at concentrations of NaCl (5% and 10%), KCl (3% and 5%), and Tween 40 (5% and 10%). Lavender flowers were soaked in these solutions before undergoing hydrodistillation, which was performed at a distillation rate of approximately 6.5 mL/min. The effect of using a hydrolat-distilled water mixture for soaking the flowers was also investigated at a distillation rate of about 6.6 mL/min.

During the hydrodistillation process, the essential oil volume was measured using the graduated tube of the Clevenger apparatus, and the time was recorded from the appearance of the first oil drop. Each experiment was conducted in duplicate.

Kinetics of essential oil hydrodistillation

The kinetics of lavender essential oil hydrodistillation was modeled using a three-parameter model [22], which posits two simultaneous processes: rapid distillation or washing of essential oil from glands on the external surface of flowers and slower diffusion of essential oil from glands within the interior of flowers:

$$y = y_s\left[1 - f \cdot e^{-k_w \cdot t} - (1-f) \cdot e^{-k_d \cdot t}\right] \quad (1)$$

where y and y_s are the essential oil yields at the time t and saturation, k_w and k_d are the rate constant of washing and diffusion, respectively, and f is the washable portion of essential oil. This model has been proven particularly effective in describing the hydrodistillation kinetics [23,24].

The initial essential oil hydrodistillation rate was calculated as the first derivative dy/dt for t = 0.

Chemical composition

The composition of the essential oil samples was performed by gas chromatography/mass spectroscopy (GC/MS) and gas chromatography/flame ionization detection (GC/FID) under conditions previously described in detail [25]. The Agilent Technologies 7890B gas chromatograph, coupled with an inert, selective 5977A mass detector with nonpolar, silica capillary column, HP-5MS (5% diphenyl- and 95% dimethyl-polysiloxane, 30 m × 0.25 mm, 0.25 μm film thickness) was used. Data analysis was performed using MSD ChemStation, MassHunter Qualitative Analysis, and AMDIS_32 software (Agilent Technologies, Santa Clara, CA, USA). The percentage composition of individual components in the essential oil was determined based on automatically integrated peak areas of the GC-FID signal.

Statistical analysis

Statistical differences were determined by one-way ANOVA followed by Tukey's HSD test, while the Kruskal-Wallis test with post-hoc Dunn's test was applied for variables that did not meet normality criteria. A significance level of 0.05 was used for all statistical analyses.

Results and discussion

Effect of lavender flower pretreatment on essential oil hydrodistillation

Figure 1 illustrates the impact of various lavender flower pretreatment methods on the hydrodistillation of essential oil using a 1000 mL flask. The distillation process involved a water-to-flower ratio of 10:1 mL/g and a distillation rate of 7.0 mL/min. It is evident that the yield of lavender essential oil increased as hydrodistillation progressed; in essence, longer hydrodistillation durations facilitated greater extraction of lavender essential oil. The pretreatment methods tested were as follows: whole (unground) flowers, wet ground flowers, dry ground (not sieved) immediately before distillation, dry ground and sieved (< 1.0 mm) immediately before distillation, and dry ground and sieved (< 1.0 mm), then stored in a paper bag in a dry, dark place until distillation.

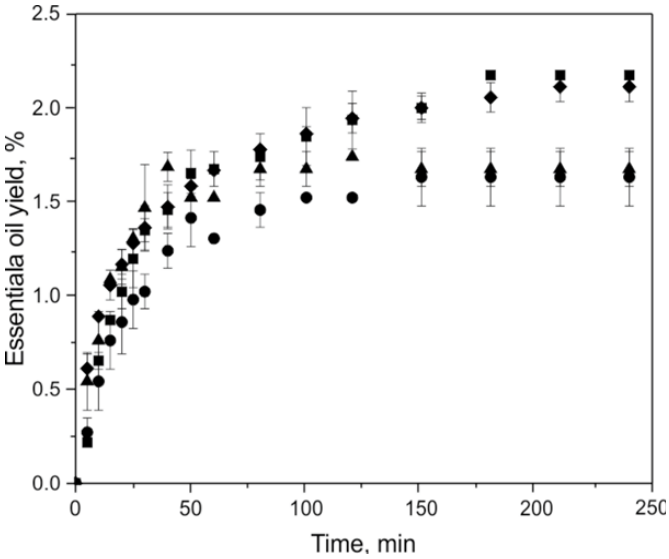

Figure 1. Effect of lavender flower pretreatment on essential oil hydrodistillation: whole (unground) – ●, dry ground, sieved, packed in a paper bag and stored in a dry, dark place until hydrodistillation – ▲, dry ground and sieved just before hydrodistillation – ▼, dry ground, not sieved, just before hydrodistillation – ■, and wet ground – ♦ (flower amount: 35 g; water-to-flower ratio: 10:1 mL/g; distillation flask volume: 1000 mL; distillation rate: 7.0 mL/min; and sieve: 1.0 mm).

Table 1 summarizes the impact of various pretreatment methods on the maximum yield of lavender essential oil and the kinetics of hydrodistillation. The maximum essential oil yield from whole (unground) flowers, wet-ground flowers, and dry-ground (not sieved) flowers just before hydrodistillation, was about 2.2%. The maximum essential oil yields from dry-ground flowers that were sieved, packed in a paper bag, and stored in a dry, dark place until hydrodistillation and dry-ground and sieved flowers just before hydrodistillation were 1.44% and 1.65%, respectively. These results showed that dry grinding and sieving of lavender flowers reduced the maximum essential oil yield by 26–35% compared to whole flowers or flowers that were dry ground immediately before distillation. Similarly, Filly *et al.* found that grinding lavender flowers before turbohydrodistillation resulted in a lower yield [16]. This reduction is likely attributed to essential oil losses during the grinding and sieving processes. The observed results can be attributed to the location of secretory glands, called trichomes, on the surface of the flowers, where they produce and store essential oils. This proximity facilitates efficient oil

extraction during processes like hydrodistillation. Labri *et al.* found that for plants with secretory sites situated near or on the surface, such as *Lavandula stoechas* and *Carum carvi*, the degree of grinding did not affect the effectiveness of hydrodistillation [26].

The washable portion of essential oil (*f*) appears to increase from 0.631 for whole flowers via 0.752 and 0.786 for wet-ground and dry-ground (but not sieved) flowers to about 0.805 for dry-ground and sieved flowers. The lowest washable portion for whole flowers was attributed to the much lower damage degree of oil-containing glands compared to ground flowers. Similarly, dry-grinding and sieving increase the initial essential oil distillation rate. The variations of the washing and diffusion rate constants with the applied pretreatment methods could not be explained although one would expect no influence of the pretreatment method. However, the two average values (k_w = 0.0867 ± 0.0221 min^{-1} and k_d = 0.0117 ± 0.0057) are statistically significantly different at $p < 0.05$ (t = 7.367 and p = 0.00004).

Table 1. Effect of lavender flower pretreatment on the maximum essential oil yield and the kinetics of hydrodistillation

Pretreatment	Initial essential oil distillation rate, %/min	F, 1	k_w, min^{-1}	k_d, min^{-1}	R^2	MRPD, %	Maximum essential oil yield ± standard deviation, %	Comparison with whole flowers, %
No pretreatment (whole flowers)	0.0573	0.631	0.0803	0.0179	0.999	± 1.5	2.22 ± 0.00	100.0
Wet grinding	0.0549	0.752	0.0707	0.0071	0.980	± 11.6	2.20 ± 0.01	99.1
Dry grinding (not sieving) just before hydrodistillation	0.0580	0.786	0.0713	0.0089	0.992	± 4.4	2.22 ± 0.02	100.0
Dry grinding and sieving just before hydrodistillation	0.0713	0.805	0.0870	0.0067	0.983	± 4.1	1.64 ± 0.03	73.9
Dry grinding, sieving, packing in a paper bag, and storing in a dry, dark place until hydrodistillation	0.0818	0.812	0.0985	0.0097	0.961	± 5.9	1.44 ± 0.00	64.9

Based on these results, using whole lavender flowers for the essential oil hydrodistillation is recommended, as it eliminates the costs associated with grinding and sieving, while also shortening the overall production time.

Effects of salt and surfactant addition

NaCl (5% and 10%), KCl (3% and 5%), and Tween 40 (5% and 10%) were chosen to assess their impact on the lavender essential oil yield and hydrodistillation rate. The previous studies have noted their beneficial effects on lavender essential oil extraction [16,19]. Certain inorganic salts have been reported to induce a 'salting-out' effect, enhancing the hydrodistillation of essential oils by increasing the polarity of the solvent [27]. This effect aids in separating hydrophilic compounds from water.

Figure 2 and Table 2 depict the influence of salt type and concentration on the hydrodistillation of the essential oil from whole lavender flowers. They indicate that the tested salts (KCl or NaCl) and their concentrations do not significantly affect the maximum yield of lavender essential oil, which averages about 1.58 ± 0.03%. Moreover, the fraction of washable components appears higher for water than for salt solutions and water (f = 0.20 ± 0.005). Similarly, the initial essential oil distillation rate is higher for water than for the salt solutions. However, the type and concentration of salts appear to influence the hydrodistillation rate by reducing the washing rate and enhancing the diffusion rate. Namely, the washing rate constant (k_w: 0.0477 ± 0,0010 min^{-1} *versus* 0.0654 min^{-1}) decreases, while the diffusion rate constant (k_d: 0,0125 ± 0,0028 min^{-1} *versus* 0.0074 min^{-1}) increases for salt solutions compared to water. Salts may disrupt oil-containing glands, facilitating easier extraction of essential oil and thereby increasing oil separation efficiency. Also, the addition of salts elevates the boiling point of the aqueous solution, thereby accelerating mass and heat transfer during essential oil hydrodistillation [28].

Filly *et al.* reported that 10% Tween 40 at hydrodistillation shows an improvement in the extraction of the essential oil from lavender (*L. hybribia* L.) flowers by about 7% compared to the conventional method, which is attributed to the reduced surface of tension between oil on the flowers and the hydrolat [16]. Also, surfactant molecules allow solubility enhancement of more hydrophobic molecules in water [29]. However, using 5% and 10% Tween 40, immediately after boiling began, the suspension in the flask began to foam and siphon upward into the Clevenger, so the experiment was terminated.

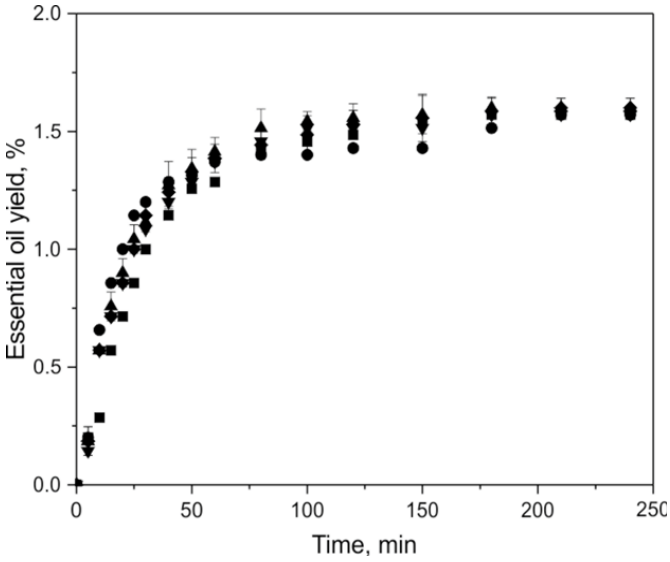

Figure 2. Effect of the type and concentration of salt on the hydrodistillation of the essential oil from whole lavender flowers: control (water, no salt) – ●; NaCl: 5% – ▲ and 10% – ▼; and KCl: 3%– ■ and 5% – ♦ (distillation flask: 1000 mL flower amount: 35 g, water-to-flower ratio: 10:1 mL/g, and distillation rate: 6.46 ± 0.17 mL/min).

Table 2. Effect of salt addition to water for soaking of lavender flowers on the maximum essential oil yield and the kinetics of hydrodistillation.

Pretreatment	Initial essential oil distillation rate, %/min	F, 1	k_w, min^{-1}	k_d, min^{-1}	R^2	MRPD, %	Maximum essential oil yield ± standard deviation, %
Water	0.0555	0.826	0.0654	0.0074	0.987	± 7.4	1.55 ± 0.03
KCl, 3%	0.0412	0.814	0.0487	0.0084	0.973	± 12.7	1.57 ± 0.00
KCl, 5%	0.0416	0.818	0.0480	0.0128	0.995	± 4.9	1.63 ± 0.00
NaCl, 5%	0.0416	0.825	0.0475	0.0144	0.996	± 4.8	1.57± 0.00
NaCl, 10%	0.0406	0.819	0.0464	0.0143	0.993	± 8.2	1.59± 0.00
		0.820 ± 0.005[b]	0.0477 ± 0.0010[a]	0.0125 ± 0.0028[a]			1.58 ± 0.03%[b]

[a] Mean value for salt solutions. [b] Mean value for water and salt solutions.

Effect of hydrolat

Figure 3 and Table 3 show the impact of using a mixture of hydrolat from the prior hydrodistillation and fresh distilled water for soaking lavender flowers on both the maximum yield of essential oil and the kinetics of the process. The maximum oil yield increased from 1.58 ± 0.02% in the initial batch, where only distilled water was used, to approximately 2.04 ± 0.10% in subsequent batches (fourth and fifth), marking a significant 29% increase. This enhancement is attributed to the extraction of additional hydrophilic compounds present in the hydrolat-water mixture, derived from the previous hydrodistillation cycles [15]. However, the use of hydrolat did not affect the washable fraction (for water f = 0.827, and for the hydrolat-water mixture f = 0.810 ± 0.019). While the use of hydrolat reduces the washing rate from 0.0654 min^{-1} for pure water to 0.0602 ± 0.0104 min^{-1} for the hydrolat-water mixture, it does substantially improve the diffusion rate from 0.0075 min^{-1} for pure water to 0.0114 ± 0.0024 min^{-1} for the mixture. Besides the washing rate, the initial essential oil distillation rate decreases in the fourth and fifth batches.

The observed phenomena can be explained by the distinct mechanisms involved in the two main stages of the essential oil hydrodistillation: washing and diffusion. Washing refers to the removal of the essential oil located on the external surface of lavender flowers, which is readily accessible and quickly removed by water during hydrodistillation. The addition of hydrolat from a previous hydrodistillation, combined with fresh distilled water, likely does not affect the solubility or accessibility of surface oil, leaving the washing rate unchanged. Diffusion, however, is the transport of the essential oil from within the plant tissues to the surface, a slower process influenced by factors such as water penetration into plant tissues and oil extraction from internal glands. The hydrolat, rich

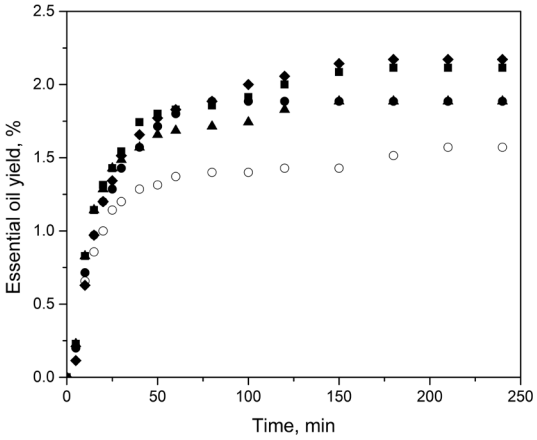

Figure 3. Effect of combining the hydrolat and fresh water on the essential oil yield from whole lavender flowers in five successive batches: the first batch (water) – o, the second batch – ●, the third batch – ▲, the fourth batch – ■, and the fifth batch – ♦ (flask volume: 1000 mL, flower amount: 35 g, hydrolat volume: 267 ± 3 mL, water-to-flower ratio: 10:1 mL/g, and distillation rate: 6.59 ± 0.12 mL/min).

Table 3. Effect of combining the hydrolat and fresh water on the maximum essential oil yield and the kinetics of hydrodistillation

No.*	Initial essential oil distillation rate, %/min	F, 1	k_w, min^{-1}	k_d, min^{-1}	R^2,	MRPD, %	Maximum essential oil yield ± standard deviation, %
I batch (water)	0.0555	0.827	0.0656	0.0074	0.987	± 7.4	1.58 ± 0.02
II batch	0.0571	0.823	0.0667	0.0127	0.978	± 11.9	1.89
III batch	0.0592	0.830	0.0689	0.0124	0.984	± 8.8	1.89
IV batch	0.0493	0.794	0.0592	0.0112	0.988	± 8.3	2.11
V batch	0.0392	0.794	0.0459	0.0133	0.987	± 18.0	1.96
		0.810 ± 0.019[a]	0.0602± 0.0104[a]	0.0114 ± 0.0024[a]			2.04 ± 0.10[b]

* I batch - Hydrodistillation using distilled water without the addition of hydrolat (control sample), batch II – using hydrolat from batch I mixed with fresh water; batch III – using hydrolat from batch II mixed with fresh water; batch IV – using hydrolat from batch III mixed with fresh water; batch V – using hydrolat from batch IV mixed with fresh water [a] Mean value for II-V batches. [b] Mean value for the IV and V batches

in previously extracted hydrophilic compounds, may enhance this process by altering the osmotic pressure between the internal plant tissues and the external solution or increasing the polarity of the solution [30-32], which may help break down oil-containing gland walls more effectively, allowing essential oil to diffuse out more easily. Also, hydrolat may act as a solvent preloaded with solubilized components from the prior hydrodistillation, which may improve the solubility of remaining compounds within the lavender flowers.

Chemical composition

Table 4 presents the chemical composition of lavender essential oils obtained under various hydrodistillation conditions, including pretreatment by different salts (KCl or NaCl) and their concentrations (3–10%), as well as the use of hydrolat from prior hydrodistillation with fresh distilled water for soaking lavender flowers over five successive batches. A total of 62 compounds were detected in all samples of essential oils, with 60 identified. The main group of identified compounds is oxidized monoterpenes (84.13–86.40%), followed by the sesquiterpenes (4.60–5.68%). As the main components in all oil samples appear linalool (30.91–33.02%), linalool acetate (16.77–20.41%), lavandulyl acetate (6.32–6.87%), while 1-octen-3-yl acetate and terpinen-4-ol were present in total percentages above 3% in all the analyzed samples.

The analysis revealed that the type of salt used during pretreatment does not have a significant impact on the majority of identified compounds in the essential oil samples. However, the concentration of certain oxygenated compounds, such as linalool and 1-octen-3-yl

Table 4. Chemical composition of the lavender essential oils obtained from the pretreated lavender flowers.a

No.	Compound	Hydrolat					NaCl		KCl	
		I batch	II batch	III batch	IV batch	V batch	5%	10%	3%	5%
		Total percent (%)					Total percent (%)		Total percent (%)	
1	n.i.	-	-	-	0.01 ± 0.01	-	-	-	-	-
2	α-Thujene	-	-	-	0.02 ± 0.01a	0.02 ± 0.01a	0.02 ± 0.01a	-	-	-
3	α-Pinene	0.08 ± 0.03ab	0.07 ± 0.07ab	0.09 ± 0.04ab	0.14 ± 0.03a	0.14 ± 0.03a	0.12 ± 0.02ab	0.10 ± 0.03ab	0.06 ± 0.05c	0.07 ± 0.02c
4	Camphene	0.08 ± 0.02a	0.10 ± 0.01a	0.08 ± 0.03a	0.11 ± 0.01a	0.10 ± 0.01a	0.06 ± 0.04a	0.06 ± 0.04a	0.09 ± 0.01a	0.07 ± 0.01a
5	n.i.	-	-	0.04 ± 0.02a	0.04 ± 0.01a	0.03 ± 0.03a	-	-	-	-
6	β-Pinene	-	0.03 ± 0.02	-	-	-	-	-	-	-
7	1-Octen-3-ol	0.05 ± 0.04a	0.06 ± 0.05a	0.06 ± 0.05a	0.06 ± 0.05a	0.05 ± 0.04a	0.06 ± 0.05a	0.05 ± 0.04a	0.05 ± 0.04a	-
8	3-Octanone	0.41 ± 0.04a	0.47 ± 0.02a	0.34 ± 0.10a	0.45 ± 0.03a	0.44 ± 0.03a	0.43 ± 0.01a	0.41 ± 0.05a	0.41 ± 0.03a	0.41 ± 0.04a
9	Myrcene	0.28 ± 0.09ab	0.32 ± 0.06a	0.26 ± 0.12ab	0.38 ± 0.08a	0.36 ± 0.07a	0.31 ± 0.05a	0.21 ± 0.02b	0.25 ± 0.05b	0.24 ± 0.06b
10	dehydroxy-*trans*-Linalool oxide	-	-	-	0.11 ± 0.09a	0.05 ± 0.04a	0.12 ± 0.10a	0.07 ± 0.05a	-	-
11	Butyl butanoate	0.15 ± 0.05a	0.16 ± 0.04a	0.14 ± 0.08a	0.13 ± 0.11a	0.20 ± 0.05a	0.13 ± 0.11a	0.15 ± 0.12a	0.15 ± 0.06a	0.15 ± 0.05a
12	3-Octanol	-	-	-	-	-	0.03 ± 0.03a	-	-	0.03 ± 0.02a
13	dehydroxy-*cis*-Linalool oxide	0.05 ± 0.01a	0.05 ± 0.01a	0.06 ± 0.02ab	0.04 ± 0.03a	0.10 ± 0.02b	0.05 ± 0.04a	0.07 ± 0.05ab	0.07 ± 0.01a	0.05 ± 0.01a
14	Hexyl acetate	0.69 ± 0.08a	0.70 ± 0.10a	0.51 ± 0.09b	0.66 ± 0.08ab	0.70 ± 0.05a	0.74 ± 0.07a	0.69 ± 0.04a	0.69 ± 0.05a	0.69 ± 0.07a
15	α-Terpinene	-	-	-	-	-	0.04 ± 0.03a	0.04 ± 0.03a	0.03 ± 0.03a	-
16	*p*-Cymene	-	-	0.03 ± 0.02a	-	0.04 ± 0.01a	0.05 ± 0.01a	0.04 ± 0.01a	0.03 ± 0.02a	-
17	o-Cymene	0.39 ± 0.00a	0.42 ± 0.04ab	0.30 ± 0.08a	0.49 ± 0.04bc	0.50 ± 0.04c	0.47 ± 0.03bc	0.46 ± 0.01b	0.40 ± 0.01a	0.36 ± 0.02a
18	Limonene	0.20 ± 0.03a	0.23 ± 0.01ab	0.12 ± 0.10a	0.24 ± 0.02ab	0.24 ± 0.02ab	0.26 ± 0.01b	0.21 ± 0.03a	0.21 ± 0.02a	0.19 ± 0.01a
19	1,8-Cineole	1.17 ± 0.03a	1.34 ± 0.10b	1.06 ± 0.10a	1.26 ± 0.07ab	1.24 ± 0.06ab	1.35 ± 0.11b	1.20 ± 0.05ab	1.23 ± 0.08b	1.15 ± 0.05ab
20	(*Z*)-β-Ocimene	0.32 ± 0.17a	0.36 ± 0.14a	0.27 ± 0.22a	0.38 ± 0.15a	0.37 ± 0.15a	0.40 ± 0.09a	0.26 ± 0.18a	0.37 ± 0.08a	0.28 ± 0.14a
21	Lavender lactone	-	-	-	0.04 ± 0.03a	0.11 ± 0.01b	-	0.07 ± 0.02a	-	-
22	(*E*)-β-Ocimene	0.18 ± 0.15a	0.21 ± 0.12a	0.19 ± 0.16a	0.24 ± 0.16a	0.25 ± 0.15a	0.25 ± 0.09a	0.15 ± 0.12a	0.16 ± 0.12a	0.15 ± 0.12a
23	γ-Terpinene	0.06 ± 0.05a	0.07 ± 0.05a	0.07 ± 0.05a	0.08 ± 0.06a	0.08 ± 0.06a	0.07 ± 0.06a	0.07 ± 0.06a	0.07 ± 0.06a	0.07 ± 0.05a
24	*cis*-Linalool oxide (furanoid)	3.31 ± 0.23a	3.64 ± 0.36a	3.33 ± 0.13a	4.20 ± 0.13b	4.30 ± 0.27b	3.34 ± 0.36a	3.69 ± 0.38a	3.28 ± 0.30a	3.53 ± 0.35a
25	*trans*-Linalool oxide (furanoid)	2.31 ± 0.31a	2.50 ± 0.20a	2.48 ± 0.11a	3.01 ± 0.25b	3.20 ± 0.22b	2.24 ± 0.14a	2.48 ± 0.21a	2.26 ± 0.14a	2.46 ± 0.19a
26	Linalool	32.32 ± 0.23a	31.90 ± 0.15a	31.47 ± 0.14b	31.42 ± 0.08b	30.91 ± 0.15c	33.02 ± 0.32d	31.58 ± 0.44ab	31.93 ± 0.19a	32.64 ± 0.17d
27	1-Octen-3-yl acetate	4.55 ± 0.33a	4.88 ± 0.01a	6.28 ± 0.04b	6.82 ± 0.04c	6.69 ± 0.01d	5.81 ± 0.16e	6.36 ± 0.02b	5.34 ± 0.20f	4.94 ± 0.01g
28	3-Octanol acetate	0.03 ± 0.02a	0.04 ± 0.03a	0.03 ± 0.02a	0.03 ± 0.02a	0.02 ± 0.01a	0.09 ± 0.02b	0.02 ± 0.02a	0.03 ± 0.02a	0.03 ± 0.02a
29	*cis*-*p*-Mentha-2,8-dien-1-ol	-	-	0.06 ± 0.05	-	-	-	-	-	-
30	Camphor	0.64 ± 0.05a	0.67 ± 0.03a	0.64 ± 0.06a	0.64 ± 0.04a	0.64 ± 0.02a	0.66 ± 0.05a	0.67 ± 0.04a	0.65 ± 0.04a	0.63 ± 0.04a
31	neo-3-Thujanol	0.08 ± 0.06a	0.08 ± 0.07a	0.10 ± 0.04a	-	0.11 ± 0.07a	-	0.12 ± 0.10a	0.10 ± 0.08a	0.07 ± 0.06a
32	Nerol oxide	0.22 ± 0.06a	0.22 ± 0.05a	0.22 ± 0.05a	0.19 ± 0.09a	0.23 ± 0.03a	0.12 ± 0.09a	0.27 ± 0.08a	0.24 ± 0.04a	0.21 ± 0.05a
33	Lavandulol	0.56 ± 0.16a	0.57 ± 0.17a	0.54 ± 0.14a	0.54 ± 0.14a	0.52 ± 0.13a	0.60 ± 0.19a	0.59 ± 0.18	0.59 ± 0.18a	0.59 ± 0.18a
34	Borneol	2.89 ± 0.53a	2.26 ± 0.67a	2.50 ± 0.89a	1.72 ± 0.10a	1.72 ± 0.34a	2.82 ± 0.68a	2.67 ± 0.86a	2.63 ± 0.85a	2.90 ± 0.54a
35	Terpinen-4-ol	3.62 ± 0.07a	3.75 ± 0.09a	3.64 ± 0.12a	3.55 ± 0.03a	3.67 ± 0.13a	3.66 ± 0.14a	3.65 ± 0.07a	3.55 ± 0.05a	3.62 ± 0.07a
36	Cryptone	1.10 ± 0.10a	1.11 ± 0.10a	1.15 ± 0.14a	1.16 ± 0.15a	1.16 ± 0.15a	1.16 ± 0.14a	1.17 ± 0.16a	1.11 ± 0.11a	1.09 ± 0.19a
37	Hexyl butanoate	0.80 ± 0.25a	0.85 ± 0.29a	0.81 ± 0.09a	0.87 ± 0.21a	0.89 ± 0.23a	0.90 ± 0.23a	0.83 ± 0.28a	0.76 ± 0.22a	0.80 ± 0.25a
38	Myrtenal	0.15 ± 0.03a	0.17 ± 0.04a	0.18 ± 0.05a	0.19 ± 0.05a	0.19 ± 0.05a	0.20 ± 0.06a	0.24 ± 0.09a	0.18 ± 0.05a	0.18 ± 0.04a
39	α-Terpineol	2.91 ± 0.01a	2.77 ± 0.10b	3.09 ± 0.11cd	3.11 ± 0.03c	2.99 ± 0.04d	3.27 ± 0.03e	3.09 ± 0.15cd	2.75 ± 0.21ab	3.00 ± 0.05d
40	Verbenone	0.16 ± 0.13a	-	0.25 ± 0.11a	0.28 ± 0.10a	0.19 ± 0.15a	0.15 ± 0.12a	0.17 ± 0.14a	0.15 ± 0.12a	0.14 ± 0.12a
41	Hydrocinnamyl alcohol	0.17 ± 0.14a	-	-	-	-	0.17 ± 0.14a	0.22 ± 0.18a	0.16 ± 0.13a	-
42	Nerol	0.12 ± 0.10	-	-	-	-	-	-	-	-
43	Ascaridole	0.35 ± 0.29a	0.34 ± 0.27a	0.34 ± 0.28a	0.41 ± 0.33a	0.38 ± 0.30a	0.32 ± 0.26a	0.33 ± 0.27a	0.31 ± 0.25a	0.32 ± 0.26a
44	Cumin aldehyde	0.23 ± 0.12a	0.18 ± 0.15a	0.27 ± 0.07a	0.28 ± 0.04a	0.18 ± 0.15a	0.33 ± 0.07a	0.21 ± 0.17a	0.20 ± 0.16a	0.23 ± 0.12a
45	Carvone	-	-	0.10 ± 0.08	-	-	-	-	-	-
46	Carvotanacetone	-	-	-	-	-	0.12 ± 0.09a	-	0.11 ± 0.09a	0.11 ± 0.09a
47	Linalool acetate	20.41 ± 0.21a	19.94 ± 0.63ab	18.60 ± 0.82bcd	16.77 ± 0.23c	16.83 ± 0.23c	17.96 ± 0.24d	19.24 ± 0.72b	20.28 ± 0.28ab	19.88 ± 0.64ab
48	Geraniol	1.14 ± 0.03a	1.07 ± 0.17a	1.11 ± 0.10a	1.13 ± 0.12a	1.14 ± 0.13a	1.22 ± 0.19a	1.13 ± 0.13a	1.17 ± 0.15a	1.18 ± 0.16a
49	*p*-Menth-1-en-7-al	0.27 ± 0.08a	0.34 ± 0.11a	0.50 ± 0.04b	0.55 ± 0.15b	0.58 ± 0.16b	0.46 ± 0.14b	0.54 ± 0.16b	0.39 ± 0.12a	0.33 ± 0.13a
50	Isobornyl acetate	0.05 ± 0.04a	0.05 ± 0.04a	0.10 ± 0.00b	0.07 ± 0.02a	0.05 ± 0.04a	0.07 ± 0.02a	-	-	0.05 ± 0.04a
51	Lavandulyl acetate	6.87 ± 0.12a	6.54 ± 0.30ab	6.78 ± 0.11a	6.49 ± 0.22ab	6.33 ± 0.26b	6.57 ± 0.11b	6.32 ± 0.32b	6.75 ± 0.20a	6.84 ± 0.34a
52	*trans*-Pinocarvyl acetate	-	-	0.17 ± 0.14a	-	0.20 ± 0.16a	-	0.19 ± 0.15a	-	-
53	Neryl acetate	0.50 ± 0.18a	0.50 ± 0.15a	0.54 ± 0.13a	0.55 ± 0.14a	0.55 ± 0.14a	0.47 ± 0.14a	0.26 ± 0.11b	0.47 ± 0.15a	0.50 ± 0.17a
54	Geranyl acetate	1.17 ± 0.02ab	1.15 ± 0.11ab	1.27 ± 0.10a	1.24 ± 0.13a	1.23 ± 0.16a	1.00 ± 0.10b	0.82 ± 0.04c	1.08 ± 0.18b	1.15 ± 0.10b
55	(*E*)-Caryophyllene	1.46 ± 0.05a	1.66 ± 0.02b	1.76 ± 0.08b	1.64 ± 0.17b	1.73 ± 0.20b	1.38 ± 0.11a	1.42 ± 0.19a	1.42 ± 0.12a	1.36 ± 0.07a
56	(*E*)-β-Farnesene	0.41 ± 0.09a	0.50 ± 0.07a	0.53 ± 0.1a5	0.49 ± 0.16a	0.52 ± 0.09a	0.42 ± 0.13a	0.47 ± 0.16a	0.44 ± 0.14a	0.40 ± 0.17a
57	Germacrene D	-	-	0.13 ± 0.10a	-	0.12 ± 0.10a	-	-	-	-
58	Lavandulyl 2-methyl butanoate	0.18 ± 0.04a	0.16 ± 0.03a	0.16 ± 0.03a	-	-	0.15 ± 0.02a	0.14 ± 0.02a	0.17 ± 0.04a	0.16 ± 0.03a
59	*cis*-Cadineneether <	-	-	-	-	-	0.10 ± 0.08a	0.09 ± 0.07a	0.11 ± 0.09a	0.12 ± 0.10a
60	*cis*-Dracunculifoliol	0.15 ± 0.12	-	-	-	-	-	-	-	-
61	Caryophyllene oxide	3.36 ± 0.39a	3.04 ± 0.17ab	3.26 ± 0.46ab	2.76 ± 0.15b	2.80 ± 0.15b	2.70 ± 0.39b	2.58 ± 0.89b	3.08 ± 0.07a	3.08 ± 0.10a
62	Khusilol	0.34 ± 0.18a	-	-	-	-	-	-	0.28 ± 0.13a	-
	Monoterpenes	1.09	1.24	0.95	1.46	1.48	1.4	1.19	1.14	1
	Oxygenated monoterpenes	85.92	85.02	85.18	84.34	84.13	85.73	85.6	85.54	86.4
	Sesquiterpenes	5.38	5.2	5.18	4.89	5.17	4.6	4.56	5.05	4.96
	Other	4.55	4.01	4.16	4.25	4.28	4.71	4.52	4.51	4.09

[a] Distillation flask volume of 1000 mL, water-to-flower ratio of 10:1 mL/g, and distillation rate of 6.5 mL/min.

* I batch - Hydrodistillation using distilled water without the addition of hydrolat (control sample), batch II – using hydrolat from batch I mixed with fresh water; batch III – using hydrolat from batch II mixed with fresh water; batch IV – using hydrolat from batch III mixed with fresh water; batch V – using hydrolat from batch IV mixed with fresh water. For unidentified compounds, the most intense m/z peaks with their relative intensities (%) are as follows: Compound 1 – m/z 43 (99.9), 72 (52.1), 127 (57.2); Compound 5 – m/z 67 (99.9), 43 (69.1), 55 (69), 58 (46.6), 141 (38.3). The values in the same row followed by the same letters did not differ significantly ($p \leq 0.5$).

acetate, was significantly influenced by salt treatment. When lavender flowers were treated with salt before distillation, the resulting slight increase in the boiling point of the aqueous solution could facilitate the effective volatilization of oil components with higher boiling points. Also, other mechanisms that can explain these effects are related to the possible formation of hydrogen bonds between salt ions and oxygenated compounds, which enhance their transfer from the plant matrix to the distillation medium, and the "salting-out effect," which decreases the solubility of oxygenated compounds in water and enhances their distillation efficiency [33]. Interestingly, the presence of Na^+ ions slightly increased the proportions of monoterpenes such as *o*- and *p*-cymene, limonene, and 1,8-cineole. In contrast, *α*-terpinene and *p*-cymene were absent in the sample obtained through hydrodistillation with distilled water, without the addition of hydrolats from the previous distillation cycles (control sample). On the other hand, linalool acetate and lavandulyl acetate were found in lower concentrations in the essential oils obtained from lavender flowers treated with NaCl compared to the control sample and oils obtained from the plant material treated with KCl. These findings suggest that the effect of different salt pretreatment on the extraction efficacy of specific compounds may be attributed to salt nature and concentration, as well as their ion properties (charge, hydration potential, solubility, ratio or types) [34]. It has also been previously confirmed

that sodium chloride can reduce the loss of heat-sensitive components [28].

The reuse of hydrolat from the previous batches in the distillation system can also affect the composition of essential oil. The addition of hydrolat, which already contains a certain amount of dissolved components from essential oil, impacts the polarity of the system, affecting the extraction process. The increased polarity further affects the solubility and volatility of essential oil compounds, impacting components of varying polarity and solubility differently. For linalool and linalyl acetate, the most abundant compounds in lavender essential oil, the addition of hydrolat reduces their concentrations. After five successive batches, the linalool content decreases slightly by 4.4%, while linalyl acetate shows a more pronounced reduction of 17.5%. The observed decrease in the concentrations of these compounds can be attributed to the addition of hydrolat, which brings hydrophilic substances, mainly oxygenated monoterpenes, from the previous distillation batch into the system. When added to distilled water for subsequent batches, these dissolved compounds may affect the diffusion and the release of oxygenated monoterpenes from the plant material, negatively impacting the yield of these compounds.

However, the proportion of *cis*- and *trans*-linalool oxide increases with the addition of hydrolats from the previous hydrodistillations, reaching 30% and 38.5%, respectively. The highest content is observed in oils obtained after reusing hydrolats from the fourth and fifth batches. Such a result suggests that the addition of hydrolats may promote oxidation conditions for linalool, leading to the formation of these products, while the concentration of linalool itself decreases. Moreover, thermolabile and oxidation-prone components, such as sesquiterpenes, may degrade due to prolonged exposure to distillation conditions. However, although caryophyllene is known to be a sensitive and oxidation-prone compound, its oxidation and degradation require temperatures above 170 °C [35]. The increased polarity due to the addition of hydrolat enhances its extraction, as caryophyllene is highly soluble in organic solvents, which may explain the higher concentration of this compound after the addition of hydrolat from the previous hydrodistillation.

Furthermore, it was observed that the essential oil contains a lower level of linalyl acetate than that prescribed by ISO standard 3515:2002 [36], according to which the content of linalyl acetate should be in the range of 25% to 47%. This deviation from the ISO standard has already been noted by several research groups [37-42,20]. These findings highlight the variability in linalool acetate concentrations, suggesting that factors such as extraction methods, and plant origin can significantly influence the chemical profile of essential oils. These differences can be explained by the process of adaptation to specific climatic and living conditions [43]. The samples of essential oils extracted from the most commonly cultivated varieties of lavender and lavandin in Serbia contain high amounts of linalool (25.2–36.9%) and linalyl acetate (6.9–28.9%) [44], which coincides with the present research. A lower concentration of linalool acetate may affect the biological effect of the essential oil. All the samples have a low camphor content, which is desirable because the oil gives off an undesirable smell [45].

Conclusion

This study aimed to optimize lavender flower pretreatment methods, assess the impact of different salt types and concentrations, and evaluate the use of hydrolat from the previous hydrodistillation batches combined with fresh distilled water on the essential oil yield and composition. Additionally, it explored the kinetics of hydrodistillation under varied process conditions.

Whole flowers were found to yield as much essential oil as wet-ground flowers, with flowers ground just before hydrodistillation also proving effective. Contrary to the previous findings, aqueous solutions of NaCl and KCl did not significantly affect the essential oil yield across concentrations from 3% to 10%. However, they did influence hydrodistillation kinetics by reducing washing rates and enhancing diffusion rates. This effect was attributed to the disruption of oil-containing glands, facilitating easier oil extraction, and increasing the boiling point of the solutions, thereby accelerating mass transfer during hydrodistillation.

Combining hydrolat from the previous batches with fresh distilled water increased the essential oil yield and improved process kinetics. While the external essential oil extraction (washing rate) remained unchanged, internal extraction (diffusion rate) was enhanced due to the hydrolat's ability to penetrate the flowers more effectively and extract oil from the interior.

The kinetics of lavender essential oil hydrodistillation was successfully described using a phenomenological model that incorporates distinct mechanisms of washing and diffusion inherent in the process.

The primary compounds identified were oxidized monoterpenes, with the two main components, linalool and linalool acetate. Pretreatment with salts and the addition of hydrolat from the previous distillation batches have distinct effects on different groups of compounds in lavender essential oils. While salt pretreatment can enhance the extraction of certain oxygenated compounds and monoterpenes, the addition of hydrolat negatively impacts the oxygenated monoterpenes extraction, affecting their solubility and volatility.

In summary, using whole lavender flowers soaked in a mixture of hydrolat from the previous hydrodistillation batches and fresh distilled water is an effective method for producing lavender essential oil. This approach minimizes energy and water consumption, thereby reducing the overall operating costs of the hydrodistillation process.

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Izvod

OPTIMIZACIJA I KINETIKA EKSTRAKCIJE ETARSKOG ULJA LAVANDE: UTICAJ PREDTRETMANA, RASTVORA SOLI I PONOVNE UPOTREBE HIDROLATA

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Cilj ovog rada bila je optimizacija predtretmana cvetova lavande, odnosno procena efekata različitih koncentracija soli i upotrebe hidrolata iz prethodne hidrodestilacije u kombinaciji sa svežom vodom na prinos i sastav etarskog ulja. Rezultati su ukazali da nesamleveni cvetovi daju istu količinu etarskog ulja kao i mokro mleveni ili sveže mleveni cvetovi. Suprotno prethodnim studijama, rastvori NaCl i KCl (3-10%) nisu imali uticaj na prinos ulja, ali su imali uticaj na kinetiku hidrodestilacije, smanjujući brzine ispiranja i povećavajući difuziju narušavanjem strukture uljnih žlezda i povećanjem tačke ključanja. Korišćenje mešavine recikliranog hidrolata i sveže vode povećalo je prinos ulja i pozitivno uticalo na kinetiku poboljšanjem ekstrakcije ulja. Proces je uspešno opisan modelom koji obuhvata mehanizme ispiranja i difuzije. Oba predtretmana cvetova lavande imala su značajan uticaj na sastav etarskog ulja. Predtretman solima je pozitivno uticao na ekstrakciju jedinjenja oksigenisanih monoterpena i monoterpena, dok je dodavanje hidrolata negativno uticalo na rastvorljivost i isparljivost oksigenisanih monoterpena. Generalno, može se zaključiti da je kvašenje celih cvetova lavande u smeši hidrolat-voda efikasan metod, koji smanjuje potrošnju energije i vode kao i operativne troškove, ali da je istovremeno neophodno obratiti pažnju na uticaj koji ovaj predtretman ima na sastav etarskog ulja.

Ključne reči: *Lavandula angustifolia,* etarsko ulje lavande, hidrodestilacija, hidrolat, kinetika destilacije.

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