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# Volatile profile in herbs of *Epilobium angustifolium*, *Epilobium hirsutum*, and *Epilobium parviflorum* growing in Estonia

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## ABSTRACT

*Epilobium* species have garnered attention for their potential use in benign prostatic hyperplasia; however, their polyphenolic composition has been primarily investigated. This study aimed to perform a comparative qualitative analysis and relative comparison based on gas chromatography–mass spectrometry (GC–MS) peak area percentages of the volatile profile in three *Epilobium* species growing in Estonia. The volatile profiles of *E. angustifolium, E. hirsutum,* and *E. parviflorum* were obtained by hydrodistillation. The yield of the volatile fraction in the studied *Epilobium* species ranged from 0.24 to 0.78 mL/kg. Qualitative and semi-quantitative analyses of the volatile profile by GC–MS revealed the presence of 98 components, with a range of 87.12% to 92.93%. The most significant proportion of the volatile profile was made up of aliphatic hydrocarbons, 40.75%–43.09% for *E. angustifolium* and 34.29%–45.04% for *E. hirsutum*, and aliphatic acids, 15.79%–29.29% for *E. angustifolium* and 7.44%–22.45% for *E. hirsutum*. *E. hirsutum* also had a significant content of aldehydes and ketones, 7.08%–9.03%. Within the volatile profile, monoterpenoids in *E. parviflorum* accounted for 51.92%, aliphatic acids for 19.43%, and sesquiterpenes for 6.76%. Thus, aliphatic hydrocarbons and fatty acids generally prevailed in *E. angustifolium* and *E. hirsutum*, while monoterpenoids predominated in *E. parviflorum.* Numerous volatile profiles (22–50) were identified for the first time in the investigated species. Although the yield of volatile profiles in *Epilobium* species is low, due to their biological activity, they may have a beneficial effect in benign prostatic hyperplasia, which requires further investigation.

## 1. Introduction

The genus willowherbs (*Epilobium* L., syn. *Chamaenerion* Ség.) of the family Onagraceae Juss. in the flora of Estonia is represented by nine species, among which the widespread ones are fireweed (*Epilobium angustifolium* L., syn. *Chamaenerion angustifolium* Scop., *Chamaenerion angustifolium* (L.) Scop.) and hairy willowherb (*Epilobium hirsutum* L.). Less commonly found in nature is hoary willowherb (*Epilobium parviflorum* Schreb.) [1]. The species are being studied in many countries because they have significant potential for biologically active substances with anti-inflammatory, antioxidant, anticancer, and antimicrobial effects [1–6].

Until now, mainly the phenolic compounds of these species have been studied. Classes of biologically active substances such as benzoic acids, phenolic acids, hydroxycinnamic acids, flavonoids, gallotannins and ellagitannins, and dimeric macrocyclic ellagitannins have been found in *E. angustifolium* [7–11], *E. hirsutum* [11–13], and *E. parviflorum* [11,14–16].

Previously, we investigated the total content of polyphenols, tannins, and flavonoids in *E. parviflorum, E. hirsutum, E. adenocaulon, E. montanum*, and *E. palustre* growing in Estonia. The content of these biologically active compounds was similar in all compared species; therefore, *E. parviflorum*, which is rarely found in nature, cannot be preferred to other species of the same genus [17]. Based on the content of

polyphenolic compounds, we concluded that *E. parviflorum* does not offer, compared to other *Epilobium* species, the best therapeutic potential for the relief of benign prostatic hyperplasia in terms of the quantitative content of these compounds [18]. The other study concluded that the most optimal, although not exclusive, collection time of *E. angustifolium* in the temperate climate zone is July–August [19].

The component composition of *E. angustifolium* essential oil has been studied the most [20–25]. The component composition of the volatile fraction from raw material samples originating from different countries differs significantly. Thus, the significant components of *E. angustifolium* volatiles from China are linoleic acid, 1-docosene, and palmitic acid [20]. The major active compounds of the essential oil of *E. angustifolium* from Poland are α-caryophyllene oxide, eucalyptol, β-linalool, camphor, (S)-carvone, and β-caryophyllene [21]. The major components of *E. angustifolium* volatiles from Lithuania are *trans*-2-hexenal, *trans*-anethole, and caryophyllenes (α- and β-) [22,23]. The major component of the essential oil of *E. angustifolium* from Turkey is limonene [24].

The major active compounds of the essential oil of *E. hirsutum* from Turkey are (Z)-3-hexene-1-ol, (Z)-3-hexenyl acetate, and cyclohexanone [26]. The most abundant component of the essential oil of *E. hirsutum* from Iran is pulegone [27]. The essential oil of *E. parviflorum* from the Czech Republic predominantly contains palmitic acid, linoleic acid, and α-linolenic acid as its primary constituents [28].

This study aimed to comparatively characterise the volatile composition of *Epilobium angustifolium, Epilobium hirsutum*, and *Epilobium parviflorum* growing in Estonia using gas chromatography–mass spectrometry (GC–MS) with the consideration of their potential pharmaceutical relevance. This study provides new comparative insights into the volatile composition of *Epilobium* species and demonstrates pronounced species-specific differences that complement existing knowledge, which is predominantly based on polyphenolic constituents.

## 2. Materials and methods

### 2.1. Plant material

The samples of the studied *Epilobium* were collected from villages in Kuusalu municipality, Harju County, Estonia (Table 1). The herbs were collected in 2025 during the flowering period (July–September) by cutting off the upper (approximately 30 cm long) flowering tops (n = 20) from the plants in dry weather. The drugs were dried in a dark, well-ventilated room at room temperature for 10 days. The dried samples were stored in closed paper bags in a dry environment, avoiding temperature fluctuations and direct light. Before distillation of the volatile profile, woody stem parts were removed from the plant material, and the resulting samples were ground into 1–3 mm particles. The Estonian plant identifier was used for species identification [29]. The species of *E. angustifolium*, *E. hirsutum*, and *E. parviflorum* belong to the same section of *Epilobium*. At the same time, some authors elevate *E. angustifolium* to the rank of the genus *Chamaenerion* [30]. The voucher specimen is stored at the Institute of Pharmacy, University of Tartu, Estonia (Nos Ona/E_ang 1, Ona/E_hir 1, and Ona/E_par 1).

### 2.2. Hydrodistillation of the volatile profile

Volatile profiles were obtained by hydrodistillation of willowherb samples using the modified method described in the *European Pharmacopoeia* monograph 'Peppermint leaf/Menthae piperitae folium' [31]. The plant materials (35 g) with 300 mL of purified water were hydrodistilled in a 1000 mL round-bottom flask for 3 hours (2–3 mL/min). Hexane (0.5 mL) was added to a graduated tube to remove the distilled oil.

### 2.3. Gas chromatography–mass spectrometry

The volatile profile samples were analysed on an Agilent 6890/5973 GC–MS system controlled by MSD ChemStation. 1 µL of the sample was introduced into the Agilent HP-5MS UI column (30 m length, 0.25 mm inner diameter, 0.25 µm film thickness) using split mode (20:1). The injector temperature was 280 °C, and the carrier gas (He) flow was kept constant at 1 mL/min throughout the whole analysis. The oven was held at 50 °C for 2 minutes, followed by a ramp of 4 °C/min to a final temperature of 280 °C and was maintained there for 5 minutes.

The MSD was operated in electron ionization (EI) mode at 70 eV, scanning across the mass range of 29–400 m/z with a delay time of 4 minutes and a scan speed of 3.8 scans/s. The data were analysed using the Agilent MassHunter software, applying a deconvolution algorithm with different window sizes. The compounds were identified by using the NIST 23 library (match factor ≥90) and by retention indices (relative to n-alkanes C8–C30) or obtained by the analysis of the reference compounds. The area percentages of each peak were calculated from the total areas in the chromatograms without using correction factors. The same GC–MS method has previously been successfully used in the analysis of other essential oils [32,33]. All GC–MS analyses were performed in triplicate, and the results are expressed as mean relative peak area (%) ± standard deviation (SD).

**Table 1.** The growing locations and essential oil yields of the studied *Epilobium* species

| Sample No. | Species | Growing place | Geographical coordinates |
|---|---|---|---|
| 1 | *E. angustifolium* (I) | Muuksi village | N59.511753 E25.520009 |
| 2 | *E. angustifolium* (II) | Uuri village | N59.510253 E25.561098 |
| 3 | *E. hirsutum* (I) | Muuksi village | N59.511180 E25.519509 |
| 4 | *E. hirsutum* (II) | Andineeme village | N59.494379 E26.458709 |
| 5 | *E. parviflorum* | Muuksi village | N59.510326 E25.506149 |

## 3. Results

The yield of the volatile fraction in the studied *Epilobium* species ranged from 0.24 to 0.78 mL/kg. In the studied samples, 98 volatile profiles were identified. In *E. angustifolium*, 45 compounds were identified in sample I and 66 compounds in sample II. In *E. hirsutum*, 63 compounds were found in sample I and 62 compounds in sample II. In *E. parviflorum*, 49 compounds were identified (Table 2).

Low standard deviations observed for most compounds indicated good analytical repeatability, while higher variability of some aliphatic acids and hydrocarbons reflected natural compositional differences between samples (Table 2). Apparent qualitative and semi-quantitative differences in volatile profiles were observed among the studied *Epilobium* species, with each species characterised by a distinct dominance of compound classes and major constituents. The studied *Epilobium* species differed in their volatile composition, with *E. angustifolium* and *E. hirsutum* dominated by long-chain aliphatic hydrocarbons and fatty acids, whereas *E. parviflorum* was characterised by a predominance of monoterpenoids, particularly carvone and menthone. Due to the descriptive and semi-quantitative nature of the GC–MS data, no formal statistical hypothesis testing was applied to individual compounds.

## 4. Discussion

The volatile fraction yield of the studied *Epilobium* species (0.24–0.78 mL/kg) was consistent with previous reports, with *E. angustifolium* showing the highest values. At the level of compound classes, *E. angustifolium* and *E. hirsutum* were dominated by aliphatic hydrocarbons and fatty acids, while *E. hirsutum* additionally exhibited a notable contribution of aldehydes and ketones.

This corresponds to the data of other researchers (Fig. 1) [20,26].

The volatile profile of *E. parviflorum* differed significantly. Monoterpenoids formed 51.92% of the essential oils of *E. parviflorum*, aliphatic acids 19.43%, and sesquiterpenes 6.76%. The obtained data regarding the significant content of aliphatic acids, monoterpenoids, carvone, and menthone in the terpenoid composition corresponded with the data from researchers in the Czech Republic [28].

A core set of 18 volatile compounds was shared among all the studied species, representing aldehydes, monoterpenes, sesquiterpenoids, apocarotenoids, fatty acids, and aliphatic hydrocarbons. The strong correlation ($r = 0.96$) observed between apocarotenoids and diterpenoid-derived compounds suggests coordinated biosynthetic pathways related to carotenoid metabolism. The dominant volatile constituents (>1%) belonged mainly to aldehydes and ketones, monoterpenoids, sesquiterpenes and sesquiterpenoids, apocarotenoids, fatty acids, and long-chain aliphatic hydrocarbons. Various types of activity have been established for most of the terpenes, terpenoids, apocarotenoids, and other groups of biologically active compounds found in *Epilobium* [34–37].

Sesquiterpenes in the studied *Epilobium* species were represented by acyclic compounds such as *(E)*-β-farnesene and α-farnesene; a bicyclic compound β-caryophyllene; naphthalene-type bicyclic compounds, such as α-copaene, (*Z*)-4(15),5-diene, and δ-cadinene; and a tricyclic compound β-copaene. The detected sesquiterpenes represented several structural subclasses, including acyclic, bicyclic, and tricyclic frameworks, with qualitative differences among species. Such structural diversity further supports species-specific terpenoid metabolism within the genus *Epilobium*.

The volatile profiles of *E. angustifolium* II and *E. parviflorum* contain β-caryophyllene and α-humulene (formerly known as α-caryophyllene), the latter being an isomer with a broken cyclobutane ring. These compounds are often found together in plants. The essential oils of *E. angustifolium* I and *E. parviflorum* contain unsaturated sesquiterpenes: (*E,E*)-α-farnesene and (*E*)-β-farnesene, as well as alcohols: farnesol (*E. angustifolium* I, *E. hirsutum* I) and nerolidol (*E. hirsutum* I and *E. hirsutum* II), which are synthesised from them in the living plant.

The studied *Epilobium* species showed clearly distinct volatile profiles. While *E. angustifolium* and *E. hirsutum* were dominated by long-chain aliphatic hydrocarbons and fatty acids, *E. parviflorum* was characterised by a predominance of monoterpenoids, particularly carvone and menthone. Notably, a substantial number of volatile profiles detected in this study have not been previously reported for the investigated species, including several terpenoids and aromatic compounds, such as camphorquinone-3-ethylene ketal, 3-(2-pentenyl)-1,2,4-cyclopentanetrione, and related constituents, highlighting the still underexplored nature of their volatile fraction.

The traditional use of *E. angustifolium*, *E. parviflorum*, and other species for the symptomatic treatment of mild urinary disorders associated with benign prostatic hyperplasia and irritative bladder, as well as for alleviating symptoms of benign prostatic enlargement or inflammation, such as nocturia, frequent daytime urination, and incomplete emptying, can be explained not only by the phenolic and polyphenolic compounds but also by the essential oils of these plants [35,38].

The well-known antitumor properties of many isoprenoid compounds include terpenes, terpenoids, apocarotenoids, and diterpenes [39].

The dominant isoprenoids in the essential oils we studied include linalool, menthone, (*E*)-p-menthan-3-one, carvone, and carvacrol; sesquiterpenes such as β-caryophyllene and α-humulene; sesquiterpenoids such as palustrol, hexahydrofarnesyl acetone, and farnesyl acetone; and apocarotenoids such as *oxo*-β-cyclocitral, *trans*-geranylacetone, and *trans*-β-ionone. Although these compounds were isolated from other plants, their antitumor properties have been described in scientific sources [39,40].

Notably, carvone, the dominant monoterpenoid in *E. parviflorum*, has been widely reported to exhibit antiproliferative and pro-apoptotic activities, supporting the potential pharmacological relevance of the observed volatile profile [41,42].

α-Linolenic acid, present at 7.47% in *E. angustifolium* I and 3.80% in *E. hirsutum* I volatile profiles, is an omega-3 fatty acid with neuroprotective, anti-inflammatory, antioxidant, and antitumor biological properties [43,44].

**Table 2.** The volatile profile in the herbs of *Epilobium angustifolium*, *Epilobium hirsutum*, and *Epilobium parviflorum* from different growing places in Estonia

| Name | RI | Library RI | *E. angustifolium* I | *E. angustifolium* II | *E. angustifolium* | *E. hirsutum* I | *E. hirsutum* II | *E. hirsutum* | *E. parviflorum* | *E. parviflorum* |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Area ± SD, %* | Area ± SD, % | mentioned in previous studies | Area ± SD, % | Area ± SD, % | mentioned in previous studies | Area ± SD, % | mentioned in previous studies |
| The yield of volatile fraction, mL/kg | | | 0.78 ± 0.03 | 0.69 ± 0.02 | | 0.24 ± 0.01 | 0.37 ± 0.02 | | 0.64 ± 0.03 | |
| Hexanal | 800 | 800 | nd | 1.06 ± 0.001 | [24] | 0.31 ± 0.023 | 0.54 ± 0.015 | – | nd | – |
| 2-Hexenal | 849 | 848 | nd | 2.48 ± 0.040 | [22] | 1.02 ± 0.117 | 0.92 ± 0.023 | [26] | nd | – |
| o-Xylene | 893 | 888 | 0.22 ± 0.206 | 0.15 ± 0.005 | – | 0.27 ± 0.116 | 0.27 ± 0.061 | – | 0.72 ± 0.136 | – |
| α-Pinene | 932 | 937 | 0.34 ± 0.019 | 0.17 ± 0.002 | [24] | 0.13 ± 0.010 | 0.14 ± 0.004 | [26,27] | 0.15 ± 0.031 | [28] |
| 2-Pentylfuran | 991 | 991 | nd | 0.54 ± 0.005 | – | 0.33 ± 0.024 | 0.53 ± 0.012 | [26] | nd | – |
| *(E,E)*-2,4-Heptadienal | 1010 | 1010 | nd | 0.38 ± 0.019 | – | 0.38 ± 0.036 | 0.67 ± 0.029 | [26] | nd | – |
| Limonene | 1030 | 1031 | nd | 0.14 ± 0.002 | [24] | nd | 0.10 ± 0.005 | [26,27] | nd | – |
| Eucalyptol | 1030 | 1030 | 0.50 ± 0.031 | nd | [21] | nd | nd | – | 0.24 ± 0.056 | [28] |
| Benzeneacetaldehyde | 1042 | 1042 | 0.32 ± 0.029 | 1.85 ± 0.035 | [22,24] | 0.67 ± 0.049 | 1.74 ± 0.029 | [26] | nd | – |
| 5-Ethenyltetrahydro-α,5-dimethyl-2-furanmethanol | 1067 | 1066 | nd | nd | – | nd | nd | – | 0.38 ± 0.080 | – |
| Linalool | 1098 | 1099 | 0.30 ± 0.018 | 1.74 ± 0.068 | [21,22,24] | 0.36 ± 0.019 | 0.64 ± 0.011 | – | 1.28 ± 0.250 | [28] |
| Nonanal | 1103 | 1104 | 0.15 ± 0.014 | 1.68 ± 0.029 | [22,24] | 0.53 ± 0.047 | 0.99 ± 0.011 | [26] | 0.21 ± 0.040 | – |
| *(E,Z)*-2,6-Nonadienal | 1152 | 1153 | nd | 0.28 ± 0.015 | – | 0.27 ± 0.022 | 0.51 ± 0.017 | – | nd | – |
| Menthone | 1152 | 1153 | 0.53 ± 0.043 | 0.30 ± 0.010 | – | 0.30 ± 0.023 | 0.54 ± 0.020 | [27] | 7.78 ± 1.369 | [28] |
| 2-Nonenal | 1158 | 1159 | 0.11 ± 0.010 | 0.30 ± 0.008 | – | 0.18 ± 0.013 | 0.31 ± 0.007 | – | nd | – |
| *(E)*-p-Menthan-3-one | 1163 | 1164 | 1.34 ± 0.079 | nd | – | nd | nd | – | 2.19 ± 0.467 | – |
| 2-Chloro-endo-bornane | 1163 | 1165 | nd | nd | – | nd | nd | – | 0.85 ± 0.184 | – |
| Terpinen-4-ol | 1175 | 1177 | nd | nd | – | nd | nd | – | 0.44 ± 0.083 | [28] |
| 1,6-Dihydrocarveol | 1195 | 1194 | nd | nd | – | nd | nd | – | 0.66 ± 0.115 | – |
| Estragole | 1197 | 1196 | 0.14 ± 0.015 | nd | [23] | 0.17 ± 0.011 | nd | – | 0.83 ± 0.170 | [28] |
| Safranal | 1198 | 1199 | nd | 0.17 ± 0.004 | – | 0.17 ± 0.010 | 0.44 ± 0.010 | – | nd | – |
| Decanal | 1204 | 1205 | 0.10 ± 0.006 | 0.60 ± 0.014 | [22,24] | 0.31 ± 0.031 | 0.53 ± 0.016 | [26] | nd | – |
| Oxo-β-cyclocitral | 1219 | 1220 | nd | 0.51 ± 0.008 | [24] | 0.53 ± 0.048 | 1.13 ± 0.028 | – | 0.53 ± 0.100 | – |
| Carvone | 1243 | 1245 | 0.26 ± 0.027 | nd | [21] | 0.16 ± 0.012 | 0.18 ± 0.006 | – | 36.68 ± 2.250 | [28] |
| Geraniol | 1253 | 1254 | nd | 0.35 ± 0.006 | [24] | nd | 0.11 ± 0.004 | – | nd | – |
| Anethole | 1284 | 1286 | nd | nd | – | nd | nd | – | 2.15 ± 0.392 | [28] |
| Neoisomenthol acetate | 1291 | 1294 | nd | nd | – | nd | nd | – | 0.73 ± 0.154 | – |
| Carvacrol | 1300 | 1299 | 1.15 ± 0.079 | nd | [21] | nd | nd | – | 0.37 ± 0.060 | [28] |
| (*E,E*)-2,4-Decadienal | 1314 | 1316 | 0.17 ± 0.014 | 0.53 ± 0.010 | – | 0.39 ± 0.032 | 0.45 ± 0.007 | – | nd | – |
| Piperitenone | 1341 | 1342 | nd | nd | – | nd | nd | – | 0.32 ± 0.054 | [28] |
| Dehydro-ar-ionene | 1352 | 1353 | nd | 0.31 ± 0.003 | – | 0.49 ± 0.037 | 0.63 ± 0.020 | – | nd | – |
| n-Capric acid | 1375 | 1370 | 2.36 ± 1.501 | 0.65 ± 0.014 | [20,24] | 0.67 ± 0.067 | nd | – | 0.16 ± 0.023 | [28] |
| Texanol | 1375 | 1374 | nd | 0.29 ± 0.006 | – | 0.32 ± 0.011 | 0.44 ± 0.245 | – | nd | – |

* Values are expressed as mean relative peak area (%) ± SD (n = 3), RI – retention indices, nd – not detected

*Continued on the next page*

**Table 2.** *Continued*

| Name | RI | Library RI | *E. angustifolium* I Area ± SD, %* | *E. angustifolium* II Area ± SD, % | *E. angustifolium* mentioned in previous studies | *E. hirsutum* I Area ± SD, % | *E. hirsutum* II Area ± SD, % | *E. hirsutum* mentioned in previous studies | *E. parviflorum* Area ± SD, % | *E. parviflorum* mentioned in previous studies |
|---|---|---|---|---|---|---|---|---|---|---|
| α-Copaene | 1385 | 1377 | nd | nd | – | nd | nd | – | 0.60 ± 0.108 | [28] |
| 2-Butyl-2-octenal | 1376 | 1379 | nd | nd | – | 0.25 ± 0.019 | 0.28 ± 0.007 | – | nd | – |
| β-Damascenone | 1385 | 1385 | 0.19 ± 0.014 | 0.58 ± 0.013 | [24] | 0.60 ± 0.031 | 0.55 ± 0.014 | [26][28] | nd | – |
| β-Bourbonene | 1385 | 1384 | nd | 0.66 ± 0.017 | [22,23] | nd | nd | – | 0.88 ± 0.166 | [28] |
| 6-Methyl-5-(1-methylethylidene)-6,8-nonadien-2-one | 1388 | 1388 | nd | 0.97 ± 0.032 | – | 0.14 ± 0.009 | 0.18 ± 0.007 | – | nd | – |
| Dodecanal | 1407 | 1408 | nd | 0.35 ± 0.004 | [24] | 0.19 ± 0.018 | 0.27 ± 0.004 | – | nd | – |
| 1-(1,1-Dimethylethyl)-4-(2-propynyloxy)-benzene | 1415 | 1416 | nd | 0.49 ± 0.013 | – | 0.29 ± 0.030 | 0.45 ± 0.007 | – | nd | – |
| β-Caryophyllene | 1420 | 1421 | nd | 1.58 ± 0.032 | [20,22–24] | 0.13 ± 0.011 | nd | [26,27] | 2.34 ± 0.406 | [28] |
| 4-(2,4,4-Trimethylcyclohexa-1,5-dienyl)-but-3-en-2-one | 1416 | 1423 | nd | 0.49 ± 0.012 | – | 0.26 ± 0.027 | 0.38 ± 0.006 | – | nd | – |
| (*E*)-Geranylacetone | 1453 | 1453 | 0.17 ± 0.009 | 0.69 ± 0.037 | [24] | 0.74 ± 0.051 | 1.52 ± 0.008 | [26] | 0.23 ± 0.047 | [28] |
| α-Humulene | 1457 | 1456 | nd | 1.52 ± 0.011 | – | nd | nd | – | 0.53 ± 0.154 | – |
| (*E*)-β-Farnesene | 1457 | 1457 | 0.12 ± 0.007 | nd | – | nd | nd | – | 0.30 ± 0.040 | [28] |
| (*Z*)-Muurola-4(15),5-diene | 1465 | 1465 | nd | nd | – | nd | nd | – | 0.44 ± 0.073 | – |
| 1-Dodecanol | 1474 | 1474 | nd | 0.41 ± 0.013 | – | 0.47 ± 0.028 | 1.39 ± 0.032 | – | nd | – |
| Germacrene D | 1478 | 1479 | nd | nd | – | nd | nd | – | 0.55 ± 0.848 | [28] |
| β-Copaene | 1430 | 1432 | nd | nd | – | nd | nd | – | 0.46 ± 0.073 | – |
| (*E*)-β-Ionone | 1487 | 1487 | 0.23 ± 0.014 | 2.18 ± 0.045 | [20,22–24] | 1.65 ± 0.140 | 6.31 ± 0.035 | [26] | 0.45 ± 0.074 | [28] |
| 5-Methyl-2-phenyl-2-hexenal | 1491 | 1491 | 0.14 ± 0.013 | nd | – | nd | nd | – | nd | – |
| (*E,E*)-α-Farnesene | 1511 | 1508 | nd | nd | – | nd | nd | – | 0.14 ± 0.021 | – |
| Tridecanal | 1510 | 1510 | nd | 0.32 ± 0.011 | – | 0.20 ± 0.010 | 0.33 ± 0.021 | – | nd | – |
| Camphorquinone-3-ethylene ketal | 1520 | 1520 | nd | 0.19 ± 0.001 | – | 0.30 ± 0.022 | 0.53 ± 0.015 | – | nd | – |
| δ-Cadinene | 1526 | 1525 | nd | 0.14 ± 0.008 | [21,22] | nd | nd | – | 0.52 ± 0.095 | – |
| 3-(2-pentenyl)-1,2,4-Cyclopentanetrione | 1526 | 1526 | 0.22 ± 0.017 | 1.74 ± 0.059 | – | 2.38 ± 0.209 | 2.26 ± 0.055 | – | 0.42 ± 0.082 | – |
| Nerolidol | 1570 | 1564 | nd | nd | – | 0.64 ± 0.060 | 0.15 ± 0.008 | – | nd | – |
| Lauric acid | 1570 | 1567 | 3.33 ± 0.272 | 0.70 ± 0.019 | [20,24] | 1.23 ± 0.103 | nd | – | 0.88 ± 0.164 | [28] |
| Hexahydrofarnesol | 1570 | 1569 | nd | nd | – | nd | 0.13 ± 0.005 | – | nd | – |
| Palustrol | 1571 | 1568 | 1.55 ± 0.145 | nd | – | nd | nd | – | nd | – |
| Spathulenol | 1581 | 1580 | 0.21 ± 0.015 | nd | – | nd | nd | – | 0.36 ± 0.065 | [28] |
| Thujopsan-2-α-ol | 1586 | 1585 | 0.13 ± 0.008 | 0.42 ± 0.004 | – | 0.18 ± 0.029 | 0.38 ± 0.079 | – | 0.47 ± 0.099 | – |
| Ledol | 1595 | 1594 | 0.19 ± 0.020 | nd | – | nd | nd | – | 0.44 ± 0.096 | – |
| γ-Gurjunenepoxide-(1) | 1607 | 1606 | 0.58 ± 0.036 | nd | – | nd | nd | – | nd | – |
| 1-Hexadecyne | 1610 | 1612 | 0.16 ± 0.158 | 0.88 ± 0.030 | – | 0.22 ± 0.010 | 0.34 ± 0.012 | – | 0.14 ± 0.027 | – |
| Tetradecanal | 1612 | 1612 | nd | 0.76 ± 0.026 | – | 0.24 ± 0.016 | 0.36 ± 0.017 | – | 0.11 ± 0.019 | – |
| o-Anisic acid pentyl ester | 1678 | 1678 | nd | 0.27 ± 0.002 | – | 0.23 ± 0.014 | 0.41 ± 0.015 | – | nd | – |

* Values are expressed as mean relative peak area (%) ± SD (n = 3), RI – retention indices, nd – not detected

*Continued on the next page*

**Table 2.** *Continued*

| Name | RI | Library RI | *E. angustifolium* I | *E. angustifolium* II | *E. angustifolium* | *E. hirsutum* I | *E. hirsutum* II | *E. hirsutum* | *E. parviflorum* | *E. parviflorum* |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Area ± SD, %* | Area ± SD, % | mentioned in previous studies | Area ± SD, % | Area ± SD, % | mentioned in previous studies | Area ± SD, % | mentioned in previous studies |
| α-Amylcinnamic alcohol | 1680 | 1680 | nd | 0.13 ± 0.008 | – | 0.13 ± 0.008 | 0.33 ± 0.014 | – | nd | – |
| 6-Methyl-1H,6H,7H-pyrrolo[2,3-c]pyridin-7-one | 1700 | 1700 | nd | 0.11 ± 0.003 | – | 0.14 ± 0.011 | 0.30 ± 0.013 | – | nd | – |
| Pentadecanal | 1714 | 1714 | nd | 0.50 ± 0.075 | – | nd | nd | – | nd | – |
| (*E,E*)-Farnesol | 1723 | 1723 | nd | 0.83 ± 0.007 | – | 0.38 ± 0.031 | nd | – | nd | – |
| Safrolglycol | 1753 | 1752 | 0.37 ± 0.021 | nd | – | nd | nd | – | nd | – |
| Myristic acid | 1770 | 1767 | 4.46 ± 1.416 | 1.03 ± 0.033 | [20,24] | 3.16 ± 0.535 | 0.28 ± 0.029 | – | 3.03 ± 1.165 | [28] |
| Isovalencenol | 1785 | 1783 | nd | 0.17 ± 0.009 | – | 0.19 ± 0.011 | 0.47 ± 0.005 | – | nd | – |
| Octadecane | 1799 | 1798 | 0.14 ± 0.018 | 0.23 ± 0.013 | [20] | nd | 0.49 ± 0.009 | – | nd | – |
| Hexadecanal | 1818 | 1815 | nd | 0.38 ± 0.022 | – | 0.15 ± 0.008 | 0.13 ± 0.013 | – | nd | – |
| 3,7,11,Trimethyl-8,10-dodecadienyl acetate | 1845 | 1839 | nd | 0.15 ± 0.005 | – | 0.30 ± 0.013 | 0.44 ± 0.041 | – | nd | – |
| Hexahydrofarnesyl acetone | 1851 | 1845 | 1.63 ± 0.162 | 0.90 ± 0.013 | [20,24] | 1.79 ± 0.178 | 5.53 ± 0.168 | [26] | 1.01 ± 0.203 | [28] |
| 1-Hexadecanol | 1889 | 1879 | 1.97 ± 0.152 | 0.55 ± 0.005 | – | 0.57 ± 0.040 | 0.64 ± 0.021 | – | nd | – |
| Nonadecane | 1909 | 1899 | 1.41 ± 0.111 | 2.23 ± 0.055 | [24] | 1.58 ± 0.155 | 0.93 ± 0.013 | [26][28] | nd | – |
| (*E,E*)-Farnesyl acetone | 1927 | 1919 | 0.29 ± 0.026 | 0.64 ± 0.021 | – | 0.67 ± 0.044 | 1.21 ± 0.010 | – | 0.19 ± 0.028 | [28] |
| Methyl palmitate | 1933 | 1925 | nd | nd | – | nd | nd | – | 0.38 ± 0.071 | [28] |
| Palmitic acid | 1990 | 1968 | 11.67 ± 5.957 | 12.44 ± 0.449 | [20,24] | 13.47 ± 6.210 | 6.90 ± 0.290 | [26] | 15.23 ± 7.540 | [28] |
| Eicosane | 2000 | 1998 | 0.36 ± 0.021 | 0.51 ± 0.031 | – | 0.54 ± 0.049 | 0.53 ± 0.009 | – | nd | – |
| Octadecanal | 2021 | 2019 | nd | 0.35 ± 0.012 | – | nd | nd | – | nd | – |
| Methyl γ-linolenate | 2076 | 2072 | 1.12 ± 0.051 | nd | – | nd | 0.16 ± 0.008 | – | nd | – |
| 6,6-Diethylhoctadecane | 2103 | 2095 | 13.82 ± 1.064 | 12.32 ± 0.332 | – | 12.11 ± 0.903 | 7.00 ± 0.123 | – | nd | – |
| α-Linolenic acid methyl ester | 2102 | 2096 | nd | nd | – | nd | nd | – | 0.52 ± 0.097 | [28] |
| Heneicosane | 2103 | 2098 | 13.80 ± 1.046 | 12.32 ± 0.335 | [20,24] | 12.12 ± 0.902 | 7.00 ± 0.125 | [26] | nd | – |
| Phytol | 2115 | 2114 | nd | 1.53 ± 0.065 | [20,24] | 4.66 ± 0.452 | 12.60 ± 0.257 | [25] | 0.27 ± 0.060 | [28] |
| Linolenic acid | 2157 | 2139 | 7.47 ± 1.148 | nd | [20] | 3.80 ± 0.744 | nd | – | nd | – |
| 1-Heneicosanol | 2428 | 2381 | nd | 0.45 ± 0.024 | – | 0.28 ± 0.013 | 0.28 ± 0.014 | – | nd | – |
| 9-octyl-Heptadecane | 2501 | 2439 | 8.60 ± 0.482 | 5.22 ± 0.209 | – | 4.05 ± 0.425 | 3.45 ± 0.062 | – | 0.35 ± 0.067 | – |
| Erucic acid | 2632 | 2543 | nd | 0.97 ± 0.006 | – | 0.12 ± 0.007 | 0.26 ± 0.007 | – | 0.13 ± 0.027 | – |
| Cyclotetracosane | 2693 | 2589 | 0.64 ± 0.051 | 4.56 ± 0.047 | – | 3.24 ± 0.274 | 1.79 ± 0.019 | – | nd | – |
| Heptacosane | 2699 | 2700 | 2.91 ± 0.226 | 1.43 ± 0.031 | – | 6.75 ± 0.509 | 7.45 ± 0.135 | – | 0.37 ± 0.024 | – |
| Heptacos-1-ene | 2836 | 2692 | nd | 1.05 ± 0.046 | – | nd | nd | – | 0.23 ± 0.043 | – |
| Nonacosane | 2898 | 2900 | 1.25 ± 0.084 | nd | – | 4.43 ± 0.287 | 5.31 ± 0.071 | – | nd | – |
| Total, % | | | 87.12 | 90.82 | | 92.93 | 91.51 | | 88.64 | |

* Values are expressed as mean relative peak area (%) ± SD (n = 3), RI – retention indices, nd – not detected

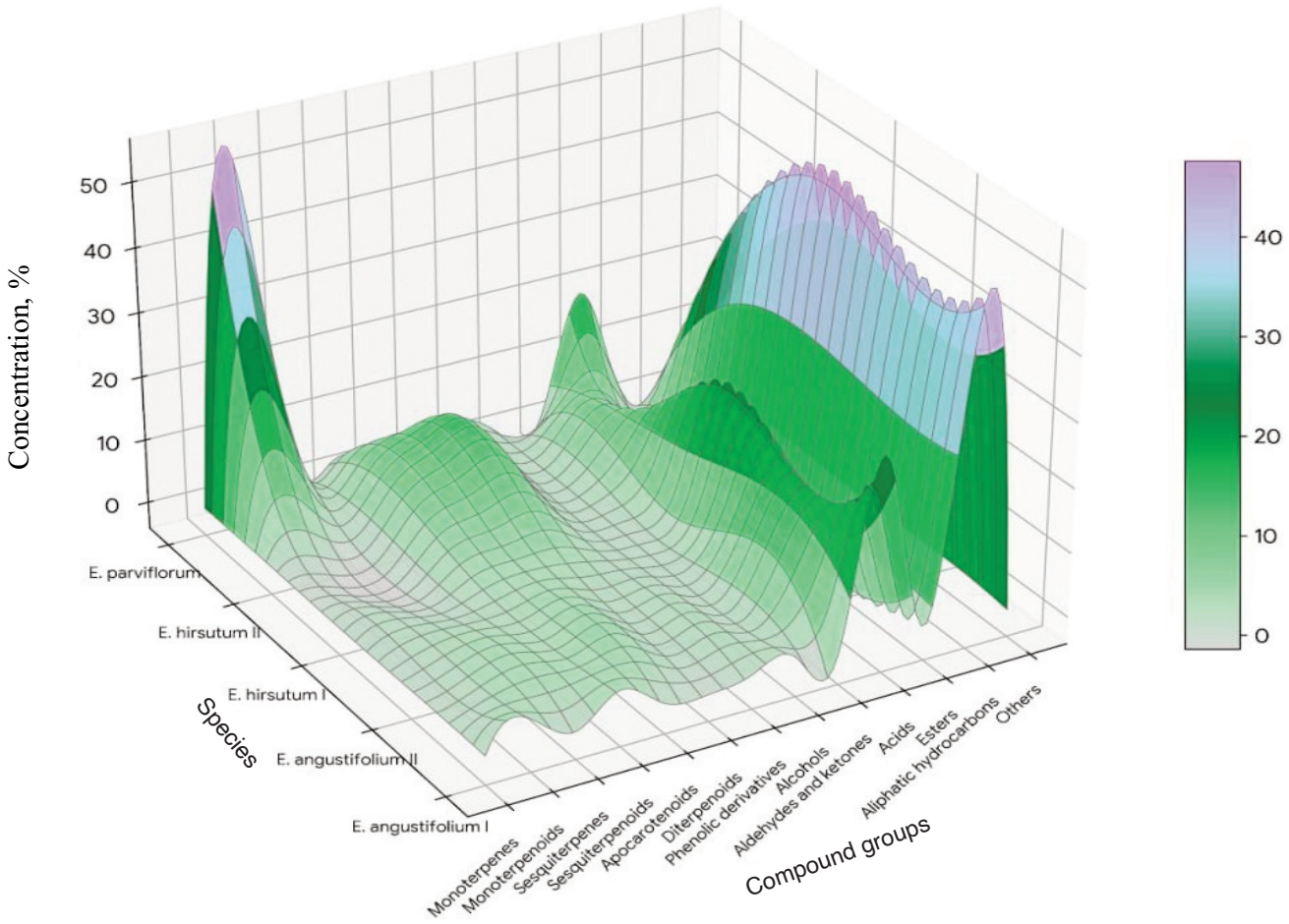


**Fig. 1.** Variation in the concentrations of the component groups of the volatile profiles of *Epilobium* species.

Although research on the pharmacological activity of agents from *Epilobium* species has traditionally focused on non-volatile polyphenols, the volatile profile (i.e. essential oil components) makes an important additional contribution to the final clinical outcome in benign prostatic hyperplasia. The volatile profile complements and enhances the primary antiproliferative effect of polyphenols, providing rapid symptom relief and eliminating the inflammatory component, thereby making *Epilobium*-based agents more effective and comprehensive in treating benign prostatic hyperplasia.

## 5. Conclusions

Overall, the examined *Epilobium* species produced volatile fractions in the range of 0.24–0.78 mL/kg, with *E. angustifolium* distinguished by the highest yield. In total, 98 volatile components were characterised in the studied samples, reflecting variations in their composition. The amounts of terpenoid compounds were the greatest in *E. parviflorum*. The main components of the volatile profiles of *E. angustifolium* and *E. hirsutum* were aliphatic hydrocarbons and aliphatic acids. Among the terpenoid compounds, the highest contents of monoterpenoids and sesquiterpenes were found in *E. parviflorum*, those of monoterpenoids and sesquiterpenoids in *E. angustifolium*, and those of diterpenoids, apocarotenoids, and sesquiterpenoids in *E. hirsutum*. The volatile profiles found may contribute to the effects of the studied plants on benign prostatic hyperplasia; however, their yield is low.

### Data availability statement

The raw, unprocessed data obtained during phytochemical analyses are available from the corresponding author upon request.

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## Eestis kasvavate *Epilobium angustifolium*, *Epilobium hirsutum* ja *Epilobium parviflorum*'i ürtide lenduvate ühendite sisaldus

**Ain Raal, Martin Lepiku, Tetiana Ilina, Alla Kovalyova, Thanh Tung Nguyen ja Oleh Koshovyi**

Pajulillede (*Epilobium*) liigid on pälvinud tähelepanu nende võimaliku kasutuse tõttu eesnäärme healoomulise suurenemise korral, kuid seni on neid uuritud peamiselt polüfenoolsete ühendite koostise seisukohast. Käesoleva töö eesmärk oli Eestis kasvavate kolme *Epilobium*'i liigi lenduvate ühendite kvalitatiivne ja suhtelisel sisaldusel põhinev võrdlev analüüs. Liikide *E. angustifolium, E. hirsutum* ja *E. parviflorum* lenduv fraktsioon eraldati taimede maapealsetest osadest destillatsiooni teel. Uuritud pajulilleliikide lenduvate fraktsioonide saagis jäi vahemikku 0,24–0,78 mL/kg. Lenduvate ühendite kvalitatiivne ja kvantitatiivne analüüs GC–MS-meetodil näitas 98 komponendi esinemist, mille osakaal moodustas 87,12–92,93% destillaadist. Suurima osa lenduvast fraktsioonist moodustasid alifaatsed süsivesinikud – *E. angustifolium*'is 40,75–43,09% ja *E. hirsutum*'is 34,29–45,04% – ning alifaatsed happed – *E. angustifolium*'is 15,79–29,29% ja *E. hirsutum*'is 7,44–22,45%. Lisaks iseloomustas *E. hirsutum*'it märkimisväärne aldehüüdide ja ketoonide sisaldus (7,08–9,03%). *E. parviflorum*'i lenduvas fraktsioonis domineerisid monoterpenoidid (51,92%), millele järgnesid alifaatsed happed (19,43%) ja seskviterpeenid (6,76%). Seega domineerivad *E. angustifolium*'i ja *E. hirsutum*'i lenduvas fraktsioonis peamiselt alifaatsed süsivesinikud ja rasvhapped, samas kui *E. parviflorum*'is on ülekaalus monoterpenoidid. Arvukalt lenduvaid ühendeid (22–50) identifitseeriti nendes taimeliikides esmakordselt. Kuigi *Epilobium*'i liikide lenduva fraktsiooni saagis on väike, võivad need ühendid tulenevalt oma bioloogilisest aktiivsusest kaasa aidata pajulillede toimele eesnäärme healoomulise suurenemise korral ning see teema vajab edasist uurimist.