Identification of Diurnal Variation of Essential Oil Components of *Hypericum perfoliatum* L. (Hypericaceae)

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Abstract
In this study, the diurnal variability of essential oil components of the species *Hypericum perfoliatum* was investigated. To determine diurnal variation, during the flowering period collection was carried out at three different times of the day (9 a.m., 12 a.m., 4 p.m.). As a result of this research, it was aimed to determine the most appropriate harvest time during the day. The essential oil components were analyzed using GC-MS device after the production of essential oil was determined by hydrodistillation method. The main components of essential oils were found to contain mainly α-pinene (41.2%), β-pinene (6.2%), n-nonane (5.6%), Y-muurolene (4.7%), α-calacorene (4.2%), δ-cadinene (3.7%), n-undecane (3.2%) and caryophyllene oxide (3.1%).

Keywords: *Hypericum*; Diurnal; Essential oil; α-pinene

Introduction
Turkey is an important gene center in terms of *Hypericum* L. (Hypericaceae) species. *Hypericum* genus, which is represented by approximately 500 species in the world, is represented by more than 100 taxa in Turkey, 44 of these taxa which are endemic [1]. *Hypericum* species have been used in the treatment of certain diseases in public medicine since the earliest times of history. In particular, *Hypericum perforatum* is successfully used by many countries to treat stomach ailments skin wounds, and especially antidepressants [2]. Due to these proven medicinal effects, it is a plant in most pharmacopoeia and monographs [3].

The development of these plants has reduced the use of all synthetic pharmaceutical industry, although synthetic drugs and herbal medicines to have effect dangerous side effects of a multifaceted, active compounds have stimulated studies on these plants and those obtained. However, the fact that it is difficult to collect from nature and the fact that the existence of its species is endangered makes it necessary to take these plants into culture [4].

Among the *Hypericum* species, hypericin and its derivatives with antifungal [5], antibacterial [6], antiviral [7] and anticancer [8] and flavonoid, floroglucinol and ksanton compounds were isolated. Hypericin and pseudohypericin are naftadiantrons with ringed structure, commonly found in *Hypericum* species and bearing numerous hydroxyl groups [4]. These molecules are often found in glands on the edges of leaves and petals [9].

The purpose of this research is to investigate the diurnal variability of essential oil components of the *Hypericum perfoliatum* species, thus determining optimal harvest times in terms of essential oil components.

Material and Method

Plant samples

*Hypericum perfoliatum* is a perennial herbaceous plant in the family Hypericaceae, spreading mainly in southern Europe, Cyprus and Africa.

The locality of the plant samples used in the study is given below. In addition, the geographical region where it was collected is presented in Figure 1. Turkey, B1 Balıkesir: from Balıkesir to Edremit 45km, roadsides, N: 39° 33.733'; E: 27° 14.902'; 252m.
During the period of full flowering plants at three different times of the day (9 a.m., 12 a.m. and 4 p.m.) biodiversity and plant samples were taken and placed in polyethylene bags, without damaging the surface from the distribution area on labeled. The collected plant material was dried in the shade at room temperature (21 °C).

**GC-MS analysis**

The determination of essential oil components of the samples was made by Thermo Electron Trace 2000 GC Model gas chromatography and Thermo Electron DSQ Quadrupole mass spectrometer in TÜBİTAK Ume Chemistry Laboratory. A homologous n-alkane series has been used as a reference in the calculation of Kovats indices (KI) (Table 1). The identification of the compounds was based on comparison of retention times and mass spectra with those obtained from the NIST and Wiley spectra from the original samples and from literature data [10].

**Table 1:** Essential oil components of *H. perfoliatum* species.

| KI* | Components          | 9a.m. (%) | 12a.m. (%) | 4p.m. (%) |
|-----|---------------------|-----------|------------|-----------|
| 900 | n-Nonane            | 5.6       | 4.7        | 4.9       |
| 939 | α-Pinene            | 41.2      | 38.9       | 38.1      |
| 953 | Camphene            | 1.2       | 2.3        | 1.6       |
| 979 | β-Pinene            | 6.2       | 4.2        | 3.8       |
| 991 | β-Myrcene           | 0.7       | 0.6        | 0.6       |
| 1022| p-Cymene            | 0.5       | 0.5        | 0.4       |
| 1029| Limonene            | 1.1       | 1.5        | 1.3       |
| 1099| n-Undecane          | 3.2       | 2.8        | 3.8       |
| 1105| n-Nonanal           | 0.6       | 0.8        | 0.6       |
| 1125| α-Campholenal       | 0.5       | 0.4        | 0.7       |
| 1377| α-Copaene           | 1.8       | 2.5        | 2.2       |
| 1420|(E)-Caryophyllene    | 2.4       | 1.8        | 2.2       |
| 1441| Aromadendrene       | 1.2       | 0.8        | 1.1       |
| 1455| α-Humulene          | 1.1       | 1          | 0.8       |
| 1468| allo-Aromadendrene  | 2.4       | 2.1        | 2.2       |
| 1481| γ-Muurolene         | 4.7       | 5.1        | 4.1       |
| 1485| Germacrene D        | 2.3       | 2.2        | 2.7       |
| 1593| Viridiflorene       | 2         | 1.2        | 2.4       |
| 1501| α-Muurolene         | 1.4       | 2          | 2.2       |
| 1513| γ-Cadinene          | 2.8       | 3.3        | 3.5       |
| 1522| δ-Cadinene          | 3.7       | 4.1        | 3.9       |
| 1543| α-Calacorene        | 4.2       | 3.8        | 4.7       |
| 1578| Spathulenol         | 2.2       | 2.8        | 2.4       |
| 1583| Caryophyllene oxide | 3.1       | 3.5        | 3.3       |
| 1625| 1-epi-Cubenol       | 0.6       | 0.5        | 0.5       |
| 1635| Cubenol             | 0.4       | 0.6        | 0.4       |
| 1657| α-Cadinol           | 0.6       | 0.5        | 0.8       |
| 1671| Cadalene            | 0.9       | 1.1        | 0.8       |
| Total| 98.6                | 95.6      | 96         |

*Kovats Index*
Findings and Discussion

Essential oil components of *H. perfoliatum* species are given in Figure 1. According to the results of essential oil components of the samples taken from the full plant at 9a.m. during the full flowering period of *H. perfoliatum* species, α-Pinene (41.2%), β-Pinene (6.2%), n-Nonane (5.6%), Y-Muurolene (4.7%), α-Calacorene (4.2%), δ-Cadinene (3.7%), n-Undecane (3.2%) and Caryophyllene oxide (3.1%) were determined.

According to the results of essential oil components of the samples taken from the whole plant at noon (12:00) during the full flowering period of *H. perfoliatum* species; α-Pinene (38.9%), Y-Muurolene (5.1%), n-Nonane (4.7%), β-Pinene (4.2%), δ-Cadinene (4.1%), α-Calacorene (3.8%), Caryophyllene oxide (3.5%) and n-Undecane (2.8%) was detected. According to the results of the essential oil components of the samples taken from the whole plant in the evening (4p.m.) at the full flowering period of *H. perfoliatum* species; α-Pinene (38.1%), n-Nonane (4.9%), α-Calacorene (4.7%), Y-Muurolene (4.1%), δ-Cadinene (3.9%), β-Pinene (3.8%), n-Undecane (3.8%) and Caryophyllene oxide (3.3%) was detected.

![Figure 1: Geographic area where the species is collected (collected region is shown with asteriks).](image)

As a result of the research, major components obtained from essential oil; α-Pinene, β-Pinene and n-Nonane were determined.

In the fat Rose (*Rosa damascena* Mill.) in their study on determination of ontogenetic, morphogenetic and diurnal variability, they found that the ratio of essential oil decreased rapidly from morning hours to evening hours and the main component rates decreased [11]. In their study on the effect of diurnal variability on yield and quality in *Thymbra capitata* genotypes, found that the most appropriate collection time in terms of essential oil ratio and components was in the morning hours [12]. They determined the most suitable collection time as noon in the species *Hypericum montbretii* Spach [13].

The essential oil composition of *Hypericum perforiatum*. As a result of their work, thirty-two compounds were identified in the essential oils of *H. perforiatum* with -pinene (13.1%), allo-aromadendrene (11.4%), germacrene-D (10.6%), n-octane (7.3%), -selinene (6.5%) and -selinene (5.5%) as main constituents. In our studies, pinene has been shown to be the major component [14].

Differences in research results are that the chemical composition of essential oil varies according to climatic, seasonal and geographical conditions [15].

Conclusion

In this study, the diurnal variability of the essential oil components of the *H. perforiatum* species was determined. The highest amount of essential oil in *H. perforiatum* was obtained from plants collected in the morning. The main components of essential oil were determined as α-Pinene, β-Pinene and n-Nonane, and it was determined that it reached the highest values in plants collected in the morning (8a.m.).

In this study, the most appropriate collection time for the essential oil components of the species examined was determined and the most effective utilization method was determined. In addition, we believe that this study will contribute to obtaining effective results in various scientific studies, especially phytochemical and pharmaceuticals, for the collection of other *Hypericum* taxa to be used other than *H. perforiatum*. 
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