Seasonal variation of phenolics content in above- and underground organs of dropwort (*Filipendula vulgaris* Moench)

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S u m m a r y

The content and chemical composition of phenolic compounds in above- and underground organs of dropwort during second year of plant vegetation were studied. Five flavonoids (hyperosid, astragalin, spireaoside, kaempferol, quercetin), 2 catechin derivatives ((+)-catechin, (-)-epigallocatechin), and 7 polyphenolic acids (ellagic, gallic, syringic, salicylic, chlorogenic, caffeic and rosmarinic) were identified in aboveground organs. Their content, both in flowers and leaves, was significantly higher at the beginning of flowering as compared with full flowering stage. In underground organs (+)-catechin and its derivatives ((-)-epigallocatechin, (-)-epigallocatechin gallate, (-)-epicatechin) as well as 2 polyphenolic acids (ellagic and gallic) were identified. Their content was not closely related to the stage of plant development.

Key words: *Filipendula vulgaris, plant development, raw materials, phenolic compounds*

INTRODUCTION

Dropwort (*Filipendula vulgaris* Moench) is a medicinal plant that has been used in folk medicine for centuries in Europe and Asia. It is a perennial of the *Rosacea* family [1]. The species occurs in small populations in dry, sunny pastures and meadows throughout most of Europe [2-5]. Flowers, herb and underground organs (rhizomes and tubers forming on the roots) are used as medicinal raw

materials since they are rich in tannins and polyphenolic acids. Flowers also contain flavonoids and essential oil [6, 7]. The raw materials reveal antibacterial, anti-inflammatory and antipyretic activity [6, 8]. In Poland, dropwort has been in use since the time before the Second World War. Nowadays, due to its decreasing occurrence, harvesting of the wild plant harvest has been abandoned [9].

The aim of our studies was to assess the yield of dropwort under cultivation conditions, and the accumulation of phenolic compounds in its aboveground and underground organs in the second year of plant vegetation.

MATERIALS AND METHODS

Studies were carried out in 2009 and 2010 on two-year-old plants cultivated at the experimental field, Warsaw University of Life Sciences (SGGW) on alluvial soil. To set up the plantation, seeds were collected in 2007 and 2008 from wild-growing population of dropwort (Podlasie region: N 52° 23.705', E 022° 53.123'). The voucher specimens of these plants are deposited at the *ex situ* collection of medicinal and aromatic plants of Warsaw University of Life Sciences. The seedlings produced in the greenhouse were planted out to the field in mid-May, 2008 and 2009 at a distance of 50 x 30 cm. The experiment was established in three replications. Flowers, leaves and underground organs of dropwort were collected at the beginning of flowering stage (early June) and at a full flowering stage (mid-June), and underground organs - again in early November (fig. 1–3). Raw materials were dried at 35°C. Fresh and air-dried mass of raw materials was calculated as a mean value of 15 plants (5 randomly chosen plants from each replication of experiment). In the air-dried material, the content and composition of phenolic compounds was determined using HPLC in three repetitions.

For phenolic compounds, the determination of 1 g of grounded raw material was extracted with 100 ml of methanol in Büchi B-811 Extraction System. After solvent evaporation, the residue was dissolved in 10 ml of methanol, filtered through a Supelco IsoDisc PTFE 25 mm × 0.45 μm filter, and subjected to HPLC for determination of phenolic compounds. The analysis was carried out using the Shimadzu chromatograph with SPD-M10A VP DAD detector. Phenomenex Luna C18 (2) 5 μm 250 × 4.6 mm column was used. Gradient elution of 10% ACN (mobile phase A) and 55% ACN (mobile phase B) (LabScan) in water adjusted to pH 3.0, flow rate 1 ml × min^{-1} and temperature 30º C was applied. Peaks were identified by comparison of retention time and spectral data with adequate parameters of standards purchased from ChromaDex. Quantification was based on the peak area at 206 nm ((-)-epigallocatechin, (+)-catechin, (-)-epicatechin, (-)-epigallocatechin gallate), 254 nm (hyperosid, quercetin) 264 nm (astragalin), 330 nm (ellagic, gallic, chlorogenic, rosmarinic, syringic, salicylic and caffeic acids) and 370 nm (spireaoside and kaempferol).

Figure 1.
Two-year-old dropwort plants in full flowering stage

Figure 2.
Flowers

Figure 3.
Underground organs – rhizomes and tuberous roots

The results were analyzed with one-way ANOVA and Tukey HSD test at the 0.05 and 0.01 significance level in Statgraphics Plus for Windows v. 4.1. The obtained results are mean value of two cycles of plant vegetation.

RESULTS AND DISCUSSION

During the second year of plant vegetation relatively high mass of both above- and underground organs was obtained. Per one plant, dry flower mass exceeded 50 g, leaves 100 g, and underground organs 300 g (tab. 1).

Table 1.

Fresh and dry mass of above- and underground organs [g · plant^{-1}]

Raw material		Beginning of flowering	Full flowering	End of vegetation
Leaves	fresh	425.2*	375.1	-
	dry	143.8	141.3	-
Flowers	fresh	260.4*	156.3	-
	dry	55.0	43.2	-
Underground organs	fresh	528.8 a	633.8 b	565.4 a
	dry	293.7 a	326.3 a	315.0 a

*$p<0.05$; values in rows marked with the same letter do not differ significantly at $\alpha=0.05$ (Tukey test)

The previous studies carried out by Radulović et al. [6], Pavlović et al. [8], Imbrea et al. [10], Smolarz et al. [11] on dropwort indicate only the presence of phenolic compounds and essential oil in the aerial parts, while in underground organs of this plant catechin derivatives and ellagic acid were found by Oszmiański [12].

In our study, among identified phenolic compounds, at the beginning of flowering and in full flowering stage, the dominating ones were spireaoside (1329.5 and 1113.6 mg · 100 g^{-1}, respectively) and hyperosid (703.0 and 516.7 mg · 100 g^{-1}, respectively) in flowers; hyperosid (678.2 and 449.9 mg · 100 g^{-1}, respectively) and chlorogenic acid (307.5 and 140.2 mg · 100 g^{-1}, respectively) in leaves (tab. 2, fig. 4, 5); and +(-)catechin (426.7 and 441.9 mg · 100 g^{-1}, respectively) in underground organs (tab. 3, fig. 6). This results indicate that the phenolic compounds content in flowers and leaves is significantly higher at the beginning of the flowering stage than during the full flowering stage. In underground organs (-)-epigallocatechin, and (-)-epigallocatechin gallate occur in the highest concentration in the early flowering plant stage, +(-) catechin – in the full flowering stage, while (-)-epicatechin and ellagic acid in late autumn. The content of gallic acid in theses organs increased to the full flowering stage, after which it noticeably decreased (tab. 3).

Table 2.

The content of phenolic compounds in aboveground organs [mg · 100 g^{-1} d. m.]

Phenolic compounds	Flowers		Leaves	
	Beginning of flowering	Full flowering	Beginning of flowering	Full flowering
Flavonoids:				
Hyperosid	703.8**	516.7	678.2**	449.9
Astragalin	546.8**	393.8	72.2	59.7
Spireaoside	1 329.5**	1 113.6	39.3*	22.3
Kaempferol	327.3**	231.3	28.8*	13.5
Quercetin	61.8	83.2*	7.6	2.8
Catechin derivatives:				
(-)-Epigallocatechin	107.2**	49.7	58.6**	33.7
(+)-Catechin	52.3	70.9	173.6**	53.8
Polyphenolic acids:				
Ellagic	301.9**	210.0	34.4	88.6**
Gallic	542.3**	477.9	152.9*	121.0
Syryngic	436.3**	344.7	262.6	334.2**
Salicylic	70.2**	46.6	28.5	21.6
Chlorogenic	0.0	0.0	307.5**	140.2
Caffeic	0.0	0.0	113.9**	68.5
Rosmarinic	0.0	0.0	132.6**	81.5

**$p<0.01$, *$p<0.05$

Table 3.

The content of phenolic compounds in underground organs [mg · 100 g^{-1} d. m.]

Phenolic compounds	Beginning of flowering	Full flowering	End of vegetation
Catechin derivatives:			
(-)-Epigallocatechin	332.1 b	276.7 a	272.1 a
(-)-Epigallocatechin gallate	155.3 b	129.8 ab	89.2 a
(+)-Catechin	426.7 ab	441.9 b	407.9 a
(-)-Epicatechin	286.4 ab	264.2 a	310.9 b
Polyphenolic acids:			
Ellagic	47.1 a	75,.9 b	89.8 b
Gallic	222.1 b	297.0 c	117.0 a

Values in rows marked with the same letter do not differ significantly at α=0.05 (Tukey test)

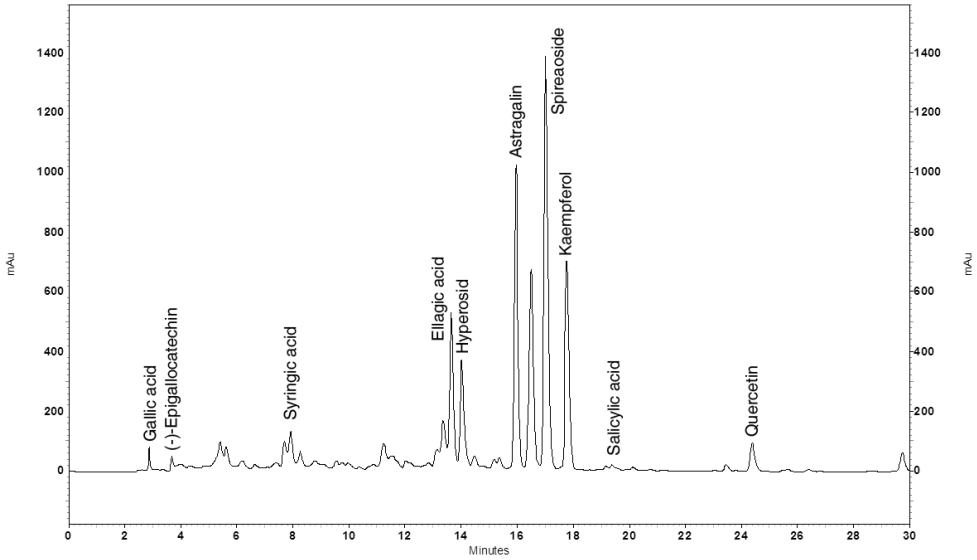

Figure 4.
HPLC chromatogram of flowers extract

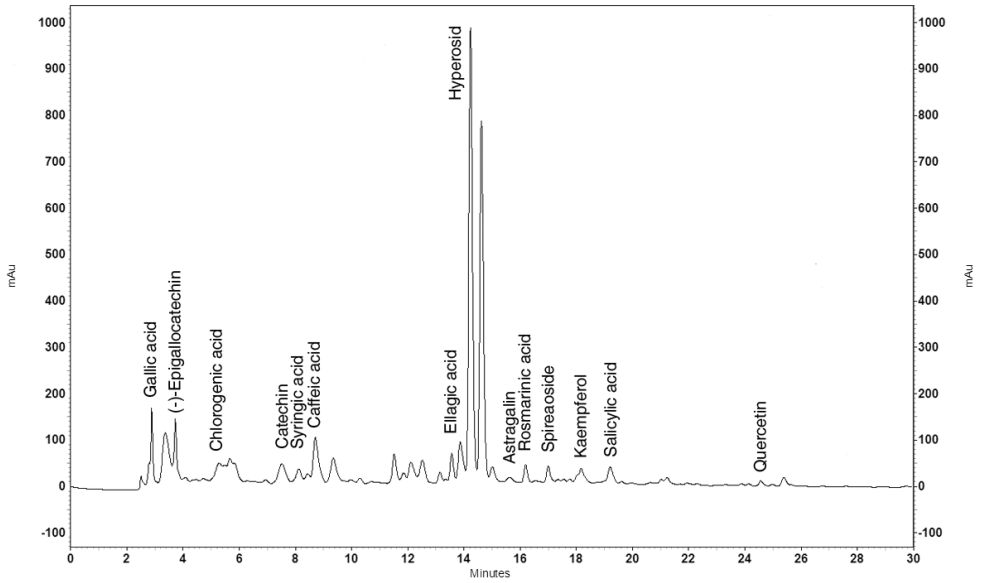

Figure 5.
HPLC chromatogram of leaves extracts

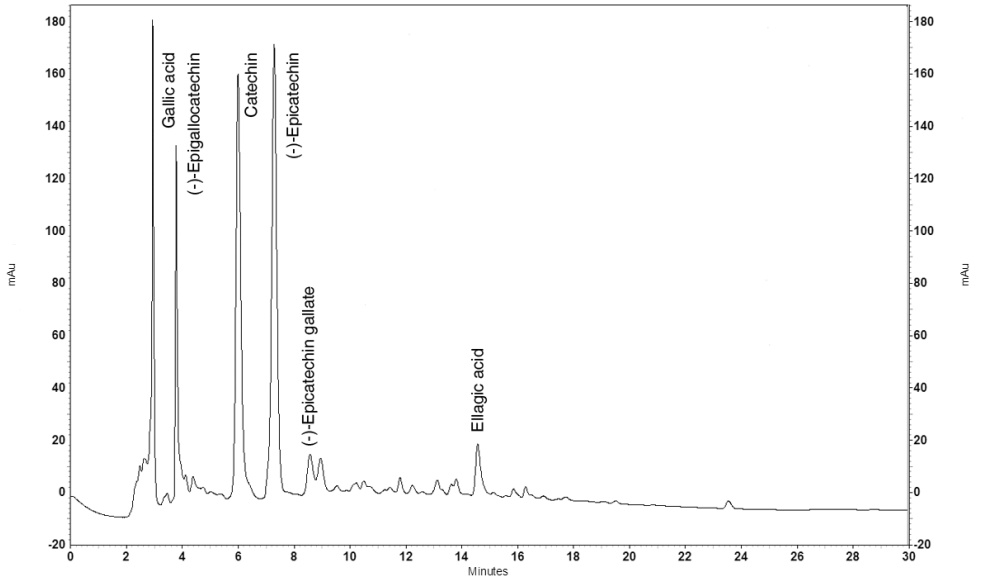

Figure 6.
HPLC chromatogram of underground organs extracts

CONCLUSIONS

- In the cultivation of dropwort it is possible to obtain high mass both of above- and underground organs.
- The content of phenolic compounds was significantly higher in flowers than in leaves.
- Dropwort flowers are characterised by a very high flavonoid content, in particular spireozide as well as phenolic acids such as ellagic, gallic and syringic.
- No clear relationship was found between the content of identified phenolic compounds in the underground organs and developmental stage of the plants.

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REFERENCES

1. Smolarz HD, Sokołowska-Woźniak A. The pharmacological activity of extracts from *Filipendula ulmaria* and *Filipendula hexapetala*. Postępy Fitoterapii 2001; 4:12-5.
2. Weidema IR, Magnussen LS, Philipp M. Gene flow and mode of pollination in a dry-grassland species, *Filipendula vulgaris* (*Rosaceae*). Heredity 2000; 84:311-20.
3. Nowiński M. Dzieje roślin i upraw leczniczych. PWRiL. Warszawa 1980.
4. Zając A., Zając M. Atlas rozmieszczenia roślin naczyniowych w Polsce. Nakładem Pracowni Chorologii Komputerowej Instytutu Botaniki UJ, Kraków 2001.
5. Matuszkiewicz W.. Przewodnik do oznaczania zbiorowisk roślinnych Polski. Warszawa 2008.
6. Radulović N, Mišić M, Aleksić J, Doković D, Palić R, Stojanović G. Antimicrobial synergism and antagonism of salicylaldehyde in *Filipendula vulgaris* essential oil. Fitoterapia 2007; 78:565-70.
7. Genig AY, Ladnaya LY. Phytochemical investigation of *Filipendula ulmaria* Maxim. and *Filipendula hexapetala* Gilib. of Lvov region flora. Farmatsevtichnii Zhurnal (Kiev) 1980; 1:50-2.
8. Pavlovic M, Petrovic S, Ristic M, Maksimovic Z, Kovacevic N. Essential oil of *Filipendula hexapetala*. Chem Nat Comp 2007; 43(2):228-9.
9. Motyka J, Panycz T. Medicinal and industrial plants in Poland. Lvov-Warsaw 1936.
10. Imbrea IM, Butnariu M, Nicolin A, Imbrea F. Determining antioxidant capacity of extracts of *Filipendula vulgaris* Moench from south-western Romania. J Food Agr Environ 2010; 8:111-6.
11. Smolarz HD, Dzido TH, Sokołowska-Woźniak A. High performance liquid chromatographic determination of flavonoids in *Filipendula hexapetala* Gilib. Acta Pol Pharm 1999; 56(2):169-72.
12. Oszmiański J, Wojdyło A, Lamer-Zarawska E, Świąder K. Antioxidant tannins from *Rosaceae* plant roots. Food Chem 2007; 100:579-83.

SEZONOWE ZMIANY ZAWARTOŚCI ZWIĄZKÓW FENOLOWYCH W NADZIEMNYCH I PODZIEMNYCH ORGANACH WIĄZÓWKI BULWKOWEJ (FILIPENDILA VULGARIS MOENCH)

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S t r e s z c z e n i e

Badano zawartość i skład chemiczny związków fenolowych w organach nadziemnych i podziemnych wiązówki bulwkowej w drugim roku uprawy. Pięć flawonoidów (hiperozyd, astragalina, spireozyd, kemferol i kwercetyna), 2 związki katechinowe ((-)-epigalokatechina i (+)-katechina) oraz 7 kwasów polifenolowych (elagowy, galusowy, syryngowy, salicylowy, chlorogenowy, kawowy i rozmarynowy) zidentyfikowano w organach nadziemnych. Zawartość tych związków zarówno w kwiatostanach, jak i w liściach była istotnie wyższa na początku kwitnienia niż podczas pełni kwitnienia roślin. W organach podziemnych zidentyfikowano natomiast (+)-katechinę i jej pochodne ((-)-epigalokatechinę, (-)-galusan epigalokatechiny i (-)-epikatechinę) oraz dwa kwasy fenolowe (galusowy i elagowy). Zawartość tych związków nie była ściśle związana z fazą rozwojową roślin.

Słowa kluczowe: *Filipendula vulgaris, rozwój rośliny, surowce, składniki fenolowe*