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Phytochemistry and inflorescences morphometry of Invasive *Solidago* L. (Goldenrods) species – valuable late autumn mellifers

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In Europe, two invasive North American species of *Solidago* L. have been detected: *S. canadensis* L. and *S. gigantea* Ait. Both species provide a stable late harvest and are valued by beekeepers for their ability to produce pollen and nectar in late fall. There is a significant correlation between the chemical components of flowers and goldenrod honey, so the study of the phytochemical composition of inflorescences (heads) seems to be very actual. The work aims to determine the total content of saccharides, phenolic compounds, and flavonoids in the heads of *S. canadensis* and *S. gigantea* for comparative evaluation of bee production quality, and also to specify morphometric differences in the heads of both species and to reveal the amplitude of their variability. The material was collected in the Moscow and Pskov Regions (Russia). The total content of the saccharides in *S. canadensis* heads was 27.33 ±0.54 %, with monosaccharides ~44–46 %. In *S. gigantea*'s heads total content of the saccharides was 1.5 times lower – 18.07 ±0.73 %, the content of monosaccharides was 7.39 ±0.15 %. The total content of phenolic compounds in the heads of *S. canadensis* was 105.36 ±1.45 mg GAE/100 g and in *S. gigantea*'s heads was 98.41 ±1.71 mg GAE/100 g. The total flavonoid content as quercetin equivalents was 58.23 ±0.17 mg QE/100 g in the heads of *S. canadensis* and 41.97 ±0.34 mg QE/100 g in *S. gigantea*'s heads. Thus, in *S. canadensis* heads the content of the total content of saccharides is 1.5 times higher, the content of phenolic compounds is 1.3 times higher, and the content of flavonoids is 1.4 times higher than in *S. gigantea*. A feature allowing the diagnosis of these species is the size of the heads: *S. canadensis* has smaller heads than *S. gigantea* (4.4 × 1.8 vs. 6.4 × 2.3 mm).

Keywords: *Solidago*, flowers, heads, saccharides, phenolic compounds, flavonoids

Introduction

In Europe, two alien North American species of *Solidago* L. of the Triplinervae section are detected: *S. canadensis* L. and *S. gigantea* Ait. (especially well when growing in homogeneous conditions of the experimental plot). The species differ in morphological characters as well. *S. canadensis* has pubescent shoots, short rhizomes, spreading panicles, and toothed leaves. *S. gigantea* has glabrous shoots (except for the panicle axis), long rhizomes, compact panicle, finely serrated or smooth-margin leaf blades (Figure 1). The species also differ in morphometric features of the heads, but no statistical

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analysis of these parameters has been carried out so far.

Both *Solidago* species are aggressive plants that actively disperse in ruderal habitats, meadows, pastures, fields, forests, roadsides, riverbanks, etc. (Vinogradova et al., 2010). Both species are recognized as invasive weeds that reduce natural biodiversity by displacing native plants (The most..., 2018). The ecological risks that both *Solidago* species pose to the environment are of great concern. However, there is another, positive value of *S. canadensis* and *S. gigantea*, manifesting themselves in their use as sources of valuable raw materials with high added value. Thus, both species provide a stable late harvest and are valued by beekeepers for their ability to produce pollen and nectar in the late fall. This variety of honey is rarely pumped because all of the nectar and pollen go to support the bee colonies and prepare them for wintering. According to the Canadian and Polish experience, goldenrod honey yields up to 150 kg per hectare – the same amount as sunflowers. Germacren D, which has not been identified in other monofloral kinds of honey, is present in goldenrod honey (Amtmann, 2009).

Although there is no complete similarity between the chemical components of *Solidago* flowers and goldenrod honey (Amtmann, 2009), there is a significant correlation. Honey absorbs the medicinal qualities of the plant from which it is collected, so the study of the phytochemical composition of inflorescences (heads) seems very actual.

The work aims to determine the total content of saccharides, phenolic compounds and flavonoids in heads of *S. canadensis* and *S. gigantea* for comparative evaluation of bee production quality, and also to specify morphometric differences in the structure of flowers and heads of both species and to reveal the amplitude of their variability.

Material and methodology

Biological material

Samples of *S. canadensis* and *S. gigantea* collected in local invasive populations in Moscow, Moscow Region, and Pskov Region were included in the study. Plants were collected during the beginning of flowering, and the collected samples were dried in bundles suspended in the air at room temperature in a shady place. Only inflorescences (heads), without panicle tips, were used in the phytochemical analysis. For comparative evaluation of morphometric traits, plants growing in the same agrophone were selected.

Studied samples:

1. *S. canadensis* (Moscow, Main Botanical garden, 55° 50‘ 21.3“ N 37° 35‘ 09.5“ E),
2. *S. gigantea* (Moscow, Main Botanical garden, 55° 50‘ 40.9“ N 37° 35‘ 31.8“ E),
3. *S. canadensis* (Moscow Region, Zvenigorod, 55° 42‘ 11.6“ N 36° 46‘ 29.6“ E),
4. *S. gigantea* (Moscow Region, Zvenigorod, 55° 42‘ 16.7“ N 36° 46‘ 29.6“ E),
5. *S. canadensis* (Pskov Region, Izborsk, 57° 43‘ 16.4“ N 27° 52‘ 11.3“ E).

Determination of total saccharides content and water-soluble carbohydrates

The content of water-soluble carbohydrates and total saccharides content were determined according to the methods “Determination of sugars by spectrophotometric method“ (OFS.1.2.3.0019.15 Gosudarstvennaya, 2015) and “Method for determining the content of water-soluble carbohydrates and starch from one sample“ (Patent RU 2406293 C1). The results were expressed in milligrams per 100 g of air-dry material.

Determination of total polyphenol content (TPC)

The total polyphenol content (TPC) was measured by the method “Method for determining the total content of phenolic compounds in plant samples“ (Patent RU 2700787 C1) using the Folin-Ciocalteu reagent. The samples were prepared and analysed as follows: 0.5 g of the plant sample was ground with 15 mL of 96 % ethanolic solution, extraction was carried out for 45 minutes in a water bath, followed by centrifugation. A quantity of 0.75 mL of each sample was mixed with 0.75 mL of the Folin-Ciocalteu reagent (diluted 5 times), 1.5 mL of 20 % (w/v) sodium carbonate, and 7 mL of distilled water. After 60 min in darkness, the absorbance at 725 nm was measured with the spectrophotometer Spekol (1300, Analytik Jena, Germany). Gallic acid (25–300 mg/L; R^2 = 0.998) was used as the standard. The results were expressed in mg/g DM gallic acid equivalent.

Determination of total flavonoid content

The total flavonoid content (TFC) was determined by the method of the GF XIII ed., (p. 332), art. “Grass of *Persicaria hydropiper* (L.) Delarbre“. Plant samples (0.5 g) were extracted with an 80 % solution of ethanolic solution for 60 minutes in a water bath with a reverse refrigerator. An aliquot of 2 mL of the sample was mixed with 1mL of 1 % (w/v) ethanolic solution

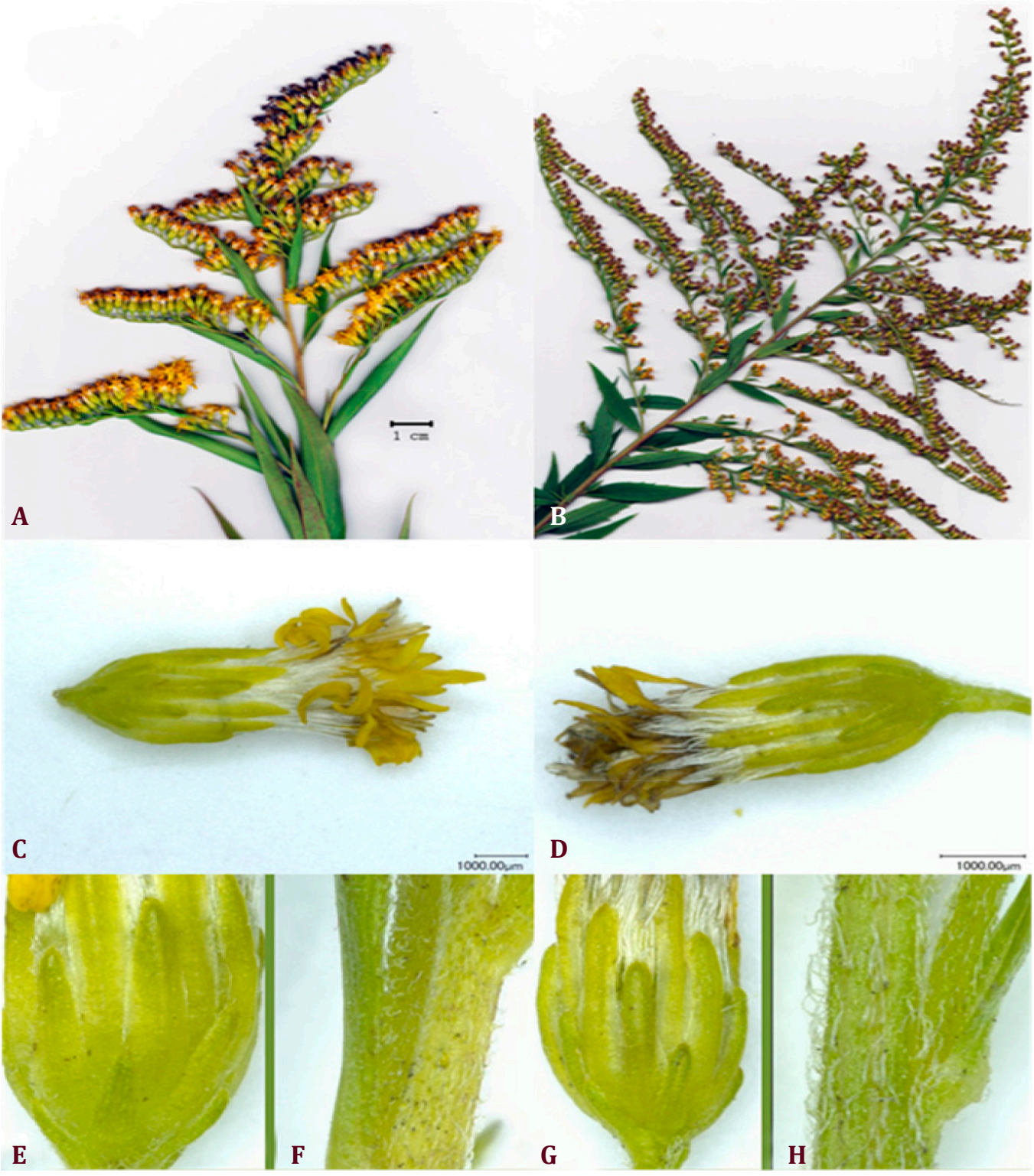

Figure 1 Morphology of generative organs of *Solidago* spp.
Panicles: A – *S. gigantea*, B – *S. canadensis*; heads: C – *S. gigantea*, D – *S. canadensis*; involucre: E – *S. gigantea*, G – *S. canadensis*; rachis: F – *S. gigantea*, H – *S. canadensis*

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of aluminum chloride and 3 mL of distilled water. After 20 min in darkness, the absorbance at 430 nm was measured using the Spekol (1300, Analytik Jena, Germany). Quercetin (1–400 mg/L; R^2 = 0.9977) was used as the standard. The results were expressed in mg/g DM quercetin equivalent.

Determination of morphometric characters

To evaluate the morphometric characters, plants growing in the same agricultural background were selected, the sample consists of 50 heads for each species; studied parameters (length and diameter of the head, length of the involucre) were measured using a digital electron microscope Keyence VHX 1000.

Statistical analysis

Basic statistical analyses were performed using PAST 2.17. Data were analysed with ANOVA test and differences between means compared through the Tukey-Kramer test ($p < 0.05$). The variability of all these parameters was evaluated using descriptive statistics.

Results and discussion

The total content of the saccharides in *S. canadensis* heads vary from 21.66 ±0.21 to 31.07 ±0.68 and the average was 27.33 ±0.54 %, with monosaccharides slightly less than half of this parameter (the average 12.05 ±0.11 %). Previously, we found that in the inflorescences of *S. canadensis*, fructose dominates in the composition of monosaccharides, all other monosaccharides were present in trace amounts (Shelepova et al., 2019a, b). In *S. gigantea* heads total content of the saccharides was 1.5 times lower – 18.07 ±0.73 %, the content of monosaccharides was 7.39 ±0.15 %. According to the literature data, goldenrods are rich in acidic polysaccharides due to the presence of hexuronic acid and its derivatives, while the presence of acidic polysaccharides and polyphenols may be the result of the aggregation of polysaccharide chains with polyphenols (Liu et al., 2018). The resulting polyphenolic glycoconjugates have anticoagulant activity, which allows us to consider them as new sources of anticoagulant compounds (Pawlaczyk et al., 2009).

A wide range of phenolic compounds is present in the inflorescences and flowers of plants. These compounds are divided into two main groups: phenolic acids and flavonoids. Therefore, one of the goals of this study was to determine the content of phenolic compounds and flavonoids in the heads of two species of goldenrod.

The total content of phenolic compounds in the heads of *S. canadensis* varied from 101.17 to 115.38 mg GAE/100 g and the average was 105.36 ±1.45 mg GAE/100 g. In *S. gigantea* heads total content of phenolic compounds was 1.3–1.1 times lower ranging from 91.04 to 101.07 mg GAE/100 g and the average was 98.41 ±1.71 mg GAE/100 g. This is significantly lower than the levels of phenolic acids (440–1200 mg/100 g) in the inflorescences of goldenrod growing in the more southern regions of Europe. But significantly higher than the levels of phenolic compounds found in honey from goldenrod (Jasicka-Misiak et al., 2018).

In the heads of *Solidago*, according to literature data, glycosides are represented mainly by glycosides of quercetin, kaempferol, and isorhamnetin (Zekič et al., 2021).

The total flavonoid content as quercetin equivalents in the heads of *S. canadensis* ranged from 49.3 to 64.1 (on average 58.2 ±0.17) mg/100 g. The highest flavonoid content (64.1 ±0.07 mg/100 g) was observed in the sample collected in Moscow, while the lowest flavonoid content (49.3 ±0.01 mg/100 g) was found in the sample collected in the Pskov region. The flavonoid content in *S. gigantea* heads was slightly lower compared to *S. canadensis* heads and averaged 41.97 ±0.34 mg/100 g.

Our results are significantly lower than the flavonoid levels found by Jasicka-Misiak et al. (2018). Thus, according to their data, the concentration of flavonoids in *Solidago* flowers was from 850 to 1 380 mg/100 g. The authors noted that the flavonoid content depends on climatic conditions. The highest levels of flavonoids were found in herbs collected in places with the greatest amount of sunlight. The authors suggest that the concentration of flavonoids in plants is proportional to the intensity of sunlight (Rosłon et al., 2014) and high temperature combined with low humidity (Karlová, 2006).

Our data on the comparison of the two species confirm the results of other studies. Thus, the analysis of flavonoids in extracts of the invasive *S. canadensis* revealed a significant increase in the content of rutin (quercetin-3-O-beta-rutinoside) in its composition compared to *S. gigantea*. The authors hypothesized that it is the synthesis of flavonoids that may determine the invasion strategy of *S. canadensis*. When entering the rhizosphere with exudates, flavonoids (mainly quercetin glycosides) can interact with ammonium. As a result, new compounds, phenol-ammonia complex, are formed. At low concentrations (20 μg/mL), these substances stimulate the formation of lateral and

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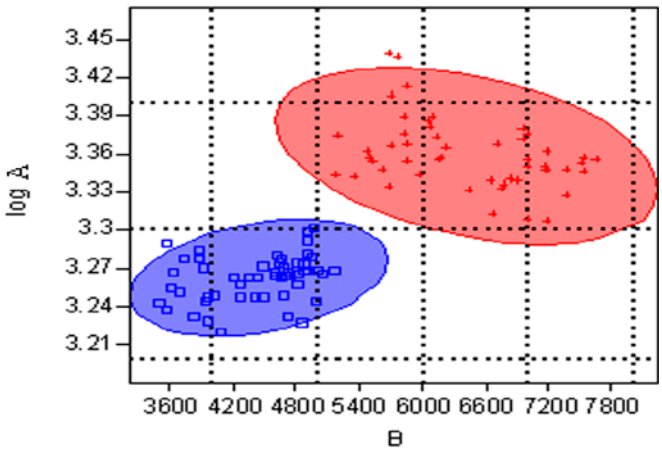

Figure 2 The head's size: *S. canadensis* (blue oval), and *S. gigantea* (red oval)
A – head's diameter; B – head's length

adventitious roots in plants, and in concentrations more than 100 µg/mL inhibit them. Enhanced synthesis and release of flavonoids, therefore, is an important element of the strategy for the biotransformation of a new habitat by alien plants species (Likhanov et al., 2021). The hypothesis about the determining influence of allelopathy on the invasive success of *S. canadensis* is also discussed by other scientists (Abhilasha et al., 2008; Li et al., 2011; Liao et al., 2011; Zhang et al., 2011; Baleţentiene, 2015; Wang et al., 2016; Mozdzen et al., 2020).

For *S. gigantea* the head's length was 5.2–7.7 mm (in average 6.4 ±0.1 mm; V = 11 %), the head's diameter 2.0–3.0 mm (2.3 ±0.0 mm; V = 9 %), the involucre's length was 2.9–4.4 mm (3.7 ±0.1 mm; V = 11 %). For *S. canadensis*, these parameters are significantly lower: the head's length was 3.5–5.2 mm (in average 4.4 ±0.1 mm; V = 11 %), the head's diameter 1.5–2.1 mm (1.8 ±0.0 mm; V = 6 %), the involucre's length was 2.6–3.9 mm (3.1 ±0.1 mm; V = 8 %). However, the relative size of the involucre, on the contrary, is higher in *S. canadensis* – it is 70 % of the head's length, while in *S. gigantea* the involucre is 60 % of the head's length.

In Central Europe, *S. canadensis* occurs more frequently than *S. gigantea* and prefers drier and warmer habitats (Kabuce and Priede, 2021). It is not advisable to intentionally cultivate *Solidago* species as melliferous plants near the apiary. In some European countries, there is even a fine for this. Both *S. canadensis* and *S. gigantea* are aggressive invasive species and can displace valuable native honey plants from natural plant communities. In this case, the bee production in the apiary will decrease during the spring and summer periods.

Conclusions

In *S. canadensis* heads the total content of saccharides is 1.5 times higher, the content of phenolic compounds is 1.3 times higher, and the content of flavonoids is 1.4 times higher than in *S. gigantea*. A feature allowing the diagnosis of these species is the size of the heads: *S. canadensis* has smaller heads than *S. gigantea* (4.4 × 1.8 vs. 6.4 × 2.3 mm).

Conflicts of Interest

The authors declare no conflict of interest.

Ethical Statement

This article does not contain any studies that would require an ethical statement.

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