

## Preliminary studies on the allelopathic potential of two invasive species of *Solidago* against selected cereal weeds

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**Abstract.** The European Union is currently pursuing a policy of moving away from synthetic plant protection products to natural ones. This is reflected in the search for natural substances that can replace them in agricultural production. Many studies indicate that invasive species of *Solidago* have such allelopathic potential. The aim of the study was to assess the effect of extracts of two *Solidago* species (*Solidago gigantea* L. and *Solidago canadensis* L.) on the growth and development of cereal weeds: *Chenopodium album*, *Papaver rhoeas* and *Agrostemma githago*. Using liquid chromatography coupled with mass spectrometry (LC - MS) techniques, the material (crude extract, phenolic and saponins fraction) was standardized and tested for allelopathic activity using the modified first generation biotest. The results indicate the phenolic fraction (20.04 mg/g dry weight of *Solidago gigantea*; 21.03 mg/g for *Solidago canadensis*) and saponin fraction (2.27 and 3.74 mg/g dry weight respectively). Both fractions from *S. gigantea* are characterized by high phytotoxicity in relation to *Ch. album*, causing over 90% reduction of its biomass in relation to the control. For *P. rhoeas* these differences are not so clear. Biomass reduction by the phenolic fraction was 20% and by the saponin fraction 50%. In the case of *S. canadensis*, allelopathic activity was not as pronounced and ranged from 20–40% for the saponin fraction relative to the *P. rhoeas*. The highest resistance to preparations made from both species of *Solidago* was demonstrated by *A. githago*.

**Key words:** allelopathy, cereal weeds, flavonoids, phenolic acids, saponins, *Solidago*

### INTRODUCTION

Originally, both species *Solidago gigantea* L. and *Solidago canadensis* L. were native to North America but were introduced into Europe as ornamental garden plants and naturalized (Semple, Cook, 2006; Woźniak et al., 2018). *Solidago* (goldenrod – English name) species were introduced into Europe in the 17th century (Weber, Jakobs, 2005). They are a perennial herbs forming large clonal colonies that tend to reduce the abundance of native biodiversity. *Solidago canadensis* and *S. gigantea* belong to the most aggressive invaders in Europe (Abhilasha et al., 2008). In many European countries, *S. gigantea* and *S. canadensis* are highly undesirable invasive species, i.e. neophytes that have a documented negative impact on the

environment and are problematic from an environmental point of view (CABI, 2019). Also in Poland *S. gigantea* and *S. canadensis* have the status of invasive species (Tokarska-Guzik et al., 2012). Both of them are an invasive species that have perfectly acclimatized in Poland and exerts strong negative pressure on native taxons. Rapid expansion of the goldenrod that causes growth in the fields, occupying more and more areas of land for agriculture and threatens native vegetation, eliminating it from ecosystems (Guzikowa, Maycock, 1986). Their success in take over new territories and new areas, this species owes to high tolerance on habitat conditions, intensive growth, production of a large amount of seeds, ease of vegetative reproduction, anemochory, lack of natural enemies (Guzikowa, Maycock, 1993), and allelopathic mechanisms (Baličević et al., 2015).



*Solidago gigantea* and *S. canadensis* have been used for centuries as medicinal plants in the areas of their original range. Herbal raw material from *Solidago* species also have been used in the European phytotherapy as a urological remedy to increase the amount of urine in cases of kidney and bladder inflammation and in irrigation therapy in urolithiasis (Woźniak et al., 2018). *Solidaginis herba* is favored due to its higher saponin and flavonoid content (Wichtl, 2013). *Solidago gigantea* and *S. canadensis* are slightly different in their phytochemical profile. The distinctive flavonoids of the aerial parts of *S. canadensis* and *S. gigantea* are rutoside (quercetin-3-rutinoside) or quercitrin (quercetin-3-O-rhamnoside), respectively (Wichtl, 2013). There are also some structural differences among triterpenes and diterpenes from the two species. *Solidago canadensis* contains mainly *trans*-clerodane and labdane-type diterpenes whereas *cis*-clerodane diterpenes are characteristic in *S. gigantea* (Woźniak et al., 2018).

A decade ago, European Union member states have changed the way they look at plant protection by introducing the concept of Integrated Plant Protection. This concept is a way of protecting plants against harmful organisms, using all available methods of plant protection, with particular reference to non-chemical methods, in such a way as to minimize the risks related to the use of plant protection products for human health and the environment (Directive 2009/128/EC). One of such possibilities may be to use allelopathic potential of plants. *Solidago* species have a proven potential to affect plants growing in the neighborhood. Bing-Yao et al. (2006) and Pisula and Meiners (2010) observed both inhibitory and stimulatory effects of *S. canadensis* against several target species. However, in higher chemical concentrations the interactions were predominantly inhibitory. In another study *S. canadensis* reduced germination of seeds in target species, but inhibition was tissue-specific, only occurring with leachates of leaves (Butcko, Jensen, 2002). The effect of allelopathic chemicals is often tested through bioassays, typically by testing the effects of plant tissue extracts on the germination of seeds (Pisula, Meiners, 2010).

The aim of this work was to show the herbicidal potential of methanol extracts and fractions containing secondary metabolites both goldenrod species against selected cereal weeds such as common poppy (*Papaver rhoeas* L.), common corn-cockle (*Agrostemma githago* L.) and goosefoot (*Chenopodium album* L.).

## MATERIALS AND METHODS

### Plant material and its preparation

Plant material for obtaining extracts was collected from plants growing in natural conditions on the fallow land in the village of Wały, Wołów county, Lower Silesian voivodeship (south-west Poland). Plants were harvested

after the elongation of shoot in phase BBCH = 39, and the obtained material was divided into above-ground parts (stem and leaves) and underground (roots and rhizomes). The underground parts were washed. Collected material was dried in the open air, obtaining an air-dry mass. The dried roots and rhizomes as well as shoots and leaves were separately ground to the material devoid of larger fragments.

Plant material was extracted three times with 7 L boiled 70% methanol (MeOH) by 1 hour. Methanol extracts were combined, concentrated and freeze-drying. We obtained 480 g of crude extract from *S. canadensis* and 512 g from *S. gigantea*. The both extracts were divided into two parts, first one crude extract (100 g) were frozen and the remaining ones were separated on a chromatographic column (100 mm × 100 mm) in reverse phase with C 18 (LiChroprep RP-18 40-63 μm), for two fractions: 40% (phenolic) and 80% MeOH (saponins). The obtained fractions were evaporated and lyophilized and then used for further studies on allelopathic activity.

### Identification and quantitative analysis

Dried root and rhizomes of *S. canadensis* and *S. gigantea* (both 300 mg) were extracted on ASE 200 Dionex, temp 100 °C, 70% MeOH into 3 x 5 min cycles. The extracts were concentrated to the aqueous phase and deposited on the Sep-Pak C18 stabilized in H<sub>2</sub>O, which was eluted with 40 and 80% MeOH to obtain the phenol fraction (40% MeOH) and saponins (80% MeOH). Collected fractions were concentrated to dryness and dissolved in 2 mL of 50% MeOH containing 0.01% HCl. Quantitative analysis was performed using a liquid chromatograph (Gilson Inc.) with a 320 PDA detector (Gilson) and ELSD (evaporative light scattering detector) detector (ELS Gilson) with column Eurosphere 100 C18 (250 x 4.6 mm, 5 μm). The chromatographic conditions were: flow rate 1 mL/min, sample injection volume of 20 μL and mobile phases A (5% MeOH, 0.01% formic acids) and B (80% MeOH, 0.01% formic acids). A gradient program was used as follows: 0% B in 5 min, from 0 to 95% B in 60 min and held for 5 min and back to 0% B in 1 min and 15 min of reconditioning before the next injection. The column temperature was maintained at 50 ± 0.1 °C. The analytical signals were monitored at 254 nm for flavonoids, 320 nm for phenolic acids. The results were calculated into rutine for flavonoids, chlorogenic acids (phenolic acids) and soya-saponine I (saponins) isolated before. Minimum limits of detection (LOD), at a signal-to-noise (S/N) ratio of 3:1, and minimum levels of quantification (LOQ), at a S/N ratio of 10:1, were determined experimentally.

For identification of the compounds used in the mass spectrometer equipped with an ion trap (Thermo LCQ Advantage MAX) connected to a Surveyor HPLC system (Thermo). The analysis used a Symmetry C18 (Waters)

(150 mm × 2.1 mm, 5 mm) chromatographic separation was performed using a linear gradient of 5–65% solvent B (acetonitrile) in solvent A (water acidified with 0.025% HCOOH) at a flow rate 0.4 mL/min. Spectrometer operated in negative ion mode while maintaining the following parameters: the potential capillary -47 V, temp. capillary 240 °C.

### Allelopathic activity

The assessment of biological activity of the tested extracts was carried out in greenhouse conditions, using the modified first generation biotest (Sekutowski, 2011) and the complete randomization method. The experiments were carried out in three cycles, in four replications. Three species of weeds were used in the experiment as acceptors: *Papaver rhoeas*, *Agrostemma githago* and *Chenopodium album*. The seeds were obtained from the wild in a fully ripe state and then stored in a cool place until the dormancy phase ended. Then the seeds were ready for use. Test plants for allelopathic studies, were germinated and then subjected to further procedures. Suitable prepared saponins fractions, phenol fractions and crude extracts were applied to test weeds in the form of spraying.

The substrate for sowing of test plants was a universal peat-mineral mix with a pH of 6.5 and sand with a diameter of 0.6–0.8 mm in a 2:1 ratio. After mixing the ingredients, pots were filled into which the weed seeds were sown. Five acceptor plants grew in each pot. When the weed plants achieved the growth stage BBCH = 12–14 (2–4 leaves), spraying with the test solutions was performed. As a control treatment for all three cycles, plants treated only with distilled water were accepted. They were then placed in a growth chamber under controlled conditions: 25 °C (± 1 °C) and 70% humidity (± 5%). After 28 days from the application of the tested solutions, the plants of each species were cut at the height of the root neck and fresh mass was determined using the analytical balance. On this basis, the loss or increase in fresh weight (in grams) was calculated compared to plants on the control treatment.

The nomenclature of the weed species names described in this paper is given for Flowering Plants and Pteridophytes of Poland – A Checklist (Mirek et al., 2002; Mirek et al., 2020).

### Preparation and application of working solutions

For the tests were used 5% and 10% working solutions of crude extracts and fractions (40% MeOH and 80% MeOH). Before use, the working solutions obtained were filtered through a filter paper, thus obtaining a homogeneous working liquid. As solvent, only distilled water was used in an amount corresponding to a dose of 250 L/ha. Working solutions for spraying were made just before the treatment. The spraying was carried out at a constant pres-

sure of 0.25 MPa, in a stationary spray chamber “Aporo”, manufactured by Przedsiębiorstwo Specjalistyczne Aporo Sp. z o.o.

### Statistical calculations and nomenclature

Statistical analysis of the results used methods of analysis of variance for experiments in a complete randomization system. The significance of the differences was tested using the Tukey half-confidence interval, and the smallest significant difference was given for a confidence level of 0.05.

Multivariate analysis of variance (MANOVA – Multivariate analysis of variance) (Morrison, 1976; Caliński, Chudzik, 1980) and discriminant analysis (Krzyśko, 1990; Mądry, 1993) were used to assess the impact of the analyzed plant extracts on weed weight. The analyzes used allowed to compare the action of extracts from the above-ground part or root of two goldenrod species in the space defined by the analyzed variables, i.e. extracts, phenolic fractions and saponin fractions. These analyzes enable correct assessment of the effects of extracts even in the case of correlation between the examined parts of the analyzed plant species.

## RESULTS AND DISCUSSION

Flavonoid glycosides were confirmed as main constituents of *Solidaginis* herba in both species which were high content of caffeoylquinic acid esters were characteristic for aerial part of this plant (Figure 1). A first major class of phenolic compounds were caffeoylquinic acid esters with the major compound in both species being 5-O-caffeoylquinic acid (neochlorogenic acid) accompanied by several mono-, di-O-caffeoylquinic and feroylquinic acids (Table 1). The composition of CQAs (caffeoylquinic acids) in aerial parts of both species was less varied than of flavonoids, but their content was approximately 3.5 times higher in *S. canadensis* (Table 1). The aerial parts of the latter species contained three major flavonols with the highest content of two rutinoides of quercetin (rutin, at 4.5 mg/g dry weight, compared to 0.1 mg/g in *S. gigantea*) and kaempferol (nicotiflorin, at 2.7 mg/g dry weight undetected in *S. gigantea*), followed by the aglycone quercetin (2.6 mg/g dry weight – two and half times higher than in *S. gigantea*). This study were confirmed by Woźniak et al. (2018). The total amount of phenolic acids were 2.8 mg/g dry weight for *S. gigantea* and 10.8 mg/g *S. canadensis*. The amount of flavonoids for *S. gigantea* was 17.24 and 10.23 mg/g for *S. canadensis*. Saponins are characteristic for this plant and total amount of saponins were 2.27 mg/g for *S. gigantea* and 3.74 mg/g for *S. canadensis*. All results were shown in Table 1.

The obtained results indicate that the crude extract (5% w/v), phenol fraction (5% w/v) and saponin fraction (10%

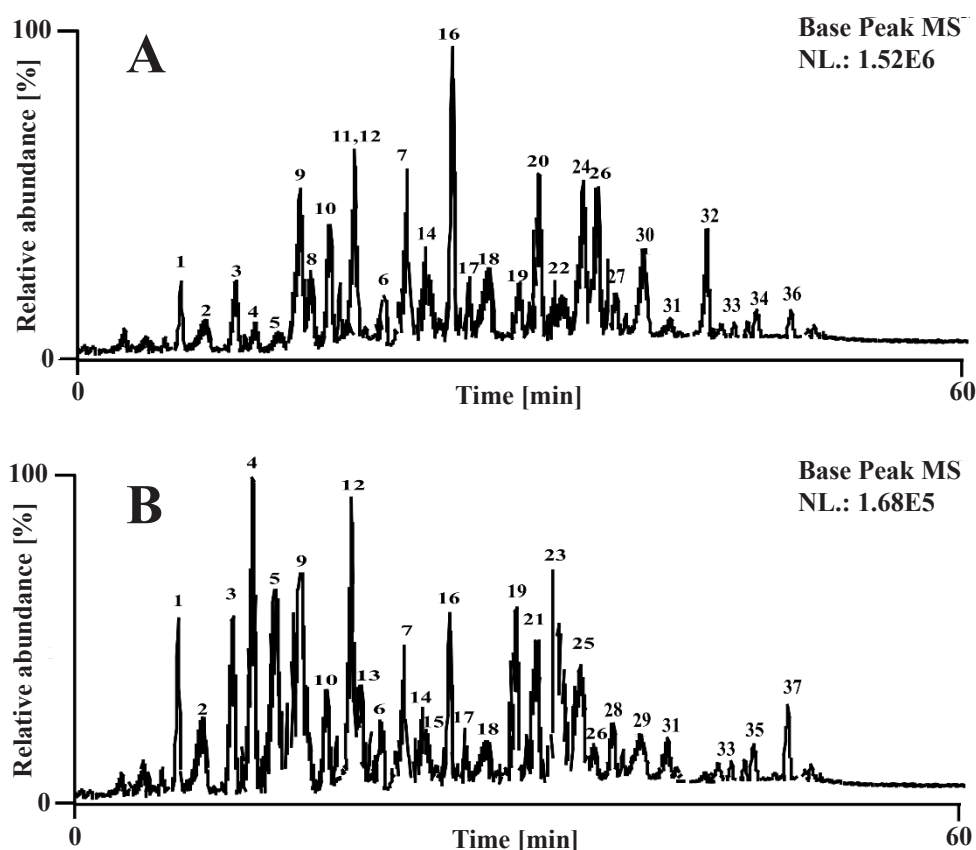


Figure 1. Chromatograms HPLC-MS base peak from aerial parts of *S. gigantea* (A) and *S. canadensis* (B).

w/v) from the *S. gigantea* are characterized by high phytotoxicity in relation to the *Ch. album*, crude extract causing over 90% reduction of its biomass in relation to the control treatment. For *P. rhoeas* biomass reduction for the phenolic fraction was about 20% and for the saponin fraction less than 50%. In the case of *S. canadensis*, allelopathic activity was not as pronounced and ranged around 20–40% for the saponin fraction relative to the *P. rhoeas*. The highest resistance to preparations made from both species was found in *A. githago*. These studies indicate a certain potential in the use of plant material as a non-toxic and fully biodegradable substitute for commonly used herbicides. All results were included in Tables 2 and 3.

Comparing the control with individual types of plant extracts, it can be concluded that the fraction of 10% of saponins to the greatest extent limited the mass of *Ch. album* regardless of the species and part of the plant. On the other hand, 5% fractions of the crude extract, phenols or saponins did not show a significant effect on *Ch. album* mass reduction (Table 4). The aerial part of *S. gigantea* showed on root and rhizomes a significantly higher herbicidal activity compared to the results obtained when using extracts from *S. canadensis*. It deserves attention is a 10% crude extract of the aerial part of the both goldenrod, which

was characterized by the highest effectiveness of reducing the weight of this weed species.

Wilks' lambda statistic for total discrimination, calculated as the ratio of the matrix of variance and intra-group covariance to the determinant of the variance and total covariance matrix, showed significant differences between the effects of extracts regardless of the species and parts of the goldenrod plants (Table 5).

Analyzing the partial Wilks' lambda, as well as the value of the F statistic for individual variable treatments, it can be shown that extracts from the *S. gigantea* regardless of the part of plants exerted a particularly large influence on the diversification of the dry matter of the analyzed weed species. On the other hand, extracts from the *S. canadensis* decreased the variability of the dry mass of *Ch. album*. The "tolerance" values – the measure of the refund of a given variable also indicate small correlations between the parts of the plants of the studied goldenrod species. The greater the distances shown in Table 6, the further apart the analyzed extracts are located in terms of the varied impact on *Ch. album* mass reduction.

When comparing the tested plant extracts with the control, significant differences between the variables examined can be found. Only the fraction of 5% saponins

Table 1. The list of compounds detected using HPLC/ESI-MS in MeOH extracts from aerial parts of *Solidago gigantea* and *Solidago canadensis*.

No.	Plant material			<i>S. gigantea</i>		<i>S. canadensis</i>
	Compound	Rt [min]	UV-VIS	[M - H] <sup>-</sup>	Amount [mg/g dry weight]	
Phenolic acids						
1	3-O-Caffeoylquinic (chlorogenic) acid	11.80	217, 323	353	0.10	0.20
2	5-O-Caffeoylquinic acid	14.40	219, 327	353	1.30	3.00
3	4-O-Caffeoylquinic acid	14.96	353, 293	353	0.10	0.10
4	1-O-Caffeoylquinic acid	17.08	353, 191	353	0.20	1.30
5	5-O-Feruloylquinic acid	18.60	325, 367	367	0.20	1.30
6	3,5-O-Dicaffeoylquinic acid	26.10	219, 327	515	0.60	4.50
7	3,4-O-Dicaffeoylquinic acid	27.50	219, 326	515	0.30	0.40
Total					2.80	10.80
Flavonoids						
8	Quercetin 3-O-rutinoside (rutin)	22.95	215, 256, 354	609	0.10	0.00
9	Quercetin-3-O-hexoside	23.49	215, 255, 354	463	1.30	0.30
10	Quercetin-O-hexoside	23.90	215, 254, 354	463	0.50	0.10
11	Kaempferol-deoxyhexoside-hexoside	24.84	218, 264, 345	593	0.04	0.00
12	Quercetin-O-pentoside	24.84	215, 255, 345	433	0.10	2.70
13	Kaempferol-O-hexoside-deoxyhexoside	25.02	218, 264, 345	593	0.00	0.30
14	Quercetin-O-pentoside	25.25	215, 255, 354	433	2.00	1.40
15	Isorhamnetin 3-O-hexoside-7-O-deoxyhexoside	25.52	215, 254, 352	623	0.00	0.03
16	Quercetin 3-O-rhamnoside	26.42	215, 259, 345	447	11.60	0.40
17	Kaempferol-O-deoxyhexoside	29.03	218, 264, 329	431	0.40	2.60
18	Quercetin	33.21	217, 255, 371	301	1.10	1.20
19	Kaempferol	42.15	218, 264, 366	285	0.10	1.20
Total					17.24	10.23
Saponins						
20	Giganteasaponin	40.30	210	1778	0.01	0.00
21	Canadensisaponin	40.30	210	1646	0.00	0.02
22	Giganteasaponin	40.80	210	970	0.40	0.00
23	Canadensisaponin	40.80	210	904	0.00	0.70
24	Canadensisaponin	41.35	210	1646	0.10	0.00
25	Canadensisaponin	41.35	210	1647	0.00	0.60
26	Canadensisaponin	42.75	210	1926	0.80	0.10
27	Canadensisaponin	43.25	210	1633	0.09	0.00
28	Canadensisaponin	43.25	210	1633	0.00	0.70
29	Canadensisaponin	43.85	210	1661	0.00	0.20
30	Giganteasaponin	45.00	210	1941	0.01	0.00
31	Giganteasaponin	46.05	210	1954	0.01	0.02
32	Canadensisaponin	46.05	210	1823	0.30	0.00
33	Canadensisaponin	46.60	210	1660	0.10	0.20
34	Giganteasaponin	48.05	210	1941	0.30	0.00
35	Canadensisaponin	48.05	210	1809	0.00	0.60
36	Giganteasaponin	48.50	210	1778	0.15	0.00
37	Canadensisaponin	48.50	210	1646	0.00	0.60
Total					2.27	3.74

RT – retention time; UV-VIS – ultraviolet-visible spectroscopy; [M - H]<sup>-</sup> – refers to an ion notation used in mass spectrometry, representing an anion formed by the deprotonation of a molecule. M – molecular mass of the analyte; -H – indicates the loss of a proton (H<sup>+</sup>) from the molecule; <sup>-</sup> signifies that the resulting ion carries a negative charge.

Table 2. The biological activity of the fractions and crude extracts from *Solidago gigantea* on production of biomass by *Chenopodium album*, *Papaver rhoeas* and *Agrostemma githago*.

Treatment	Concentration [% w/v]	Biomass production					
		<i>Ch. album</i>		<i>P. rhoeas</i>		<i>A. githago</i>	
		g	%	g	%	g	%
Control	-	6.43	100.0	5.33	100.0	10.43	100.0
Crude extract	10%	4.80	74.6	4.03	75.6	10.67	102.2
	5%	0.70	10.9	4.43	83.1	10.50	100.6
Phenolic fraction	10%	4.57	71.0	4.40	82.5	10.77	103.2
	5%	0.43	6.7	4.50	84.4	13.27	127.2
Saponin fraction	10%	0.43	6.7	3.50	65.6	10.87	104.2
	5%	4.93	76.7	2.73	51.3	11.40	109.3
LSD (0.05)		2.129		1.792		n.d.	

n.d. – no statistical differences

Table 3. The biological activity of the fractions and crude extracts from *Solidago canadensis* on production of biomass by *Chenopodium album*, *Papaver rhoeas* and *Agrostemma githago*.

Treatment	Concentration [% w/v]	Biomass production					
		<i>Ch. album</i>		<i>P. rhoeas</i>		<i>A. githago</i>	
		g	%	g	%	g	%
Control	-	6.43	100.0	5.33	100.0	10.43	100.0
Crude extract	10%	3.80	59.1	3.33	62.5	9.00	86.3
	5%	6.10	94.8	3.83	71.9	9.23	88.5
Phenolic fraction	10%	3.97	61.7	3.87	72.5	8.53	81.8
	5%	4.30	66.8	3.70	69.4	9.90	94.9
Saponin fraction	10%	4.90	76.2	3.50	65.6	7.93	76.0
	5%	5.80	90.2	4.07	76.3	8.47	81.2
LSD (0.05)		2.072		1.793		n.d.	

n.d. – no statistical differences

Table 4. Average dry matter of *Chenopodium album* depending on the applied extract from selected parts of plants of *Solidago gigantea* and *S. canadensis*.

Type of extract	<i>S. canadensis</i> root	<i>S. canadensis</i> aerial part	<i>S. gigantea</i> root	<i>S. gigantea</i> aerial part	Average
Control	8.20	8.20	8.20	8.20	8.20
Crude extract – 5%	8.53	7.43	8.60	5.93	7.62
Crude extract – 10%	7.03	7.37	7.70	3.73	6.46
Phenolic fraction – 5%	8.77	8.10	6.03	7.23	7.53
Phenolic fraction – 10%	6.80	7.90	5.70	6.37	6.69
Saponin fraction – 5%	8.07	7.87	8.53	6.33	7.70
Saponin fraction – 10%	7.53	7.00	4.70	6.03	6.32
Average	7.79	7.61	5.94	7.05	7.05

LSD *Solidago* species = 0.71; LSD types of extracts = 1.18LSD interaction (parts of plant of *Solidago* species) × (types of extracts) = 1.68

Table 5. Results of discriminant function analysis for *Chenopodium album*, treated with extracts from roots and aerial parts of *Solidago* species (*S. canadensis*, *S. gigantea*).

Wilks' lambda = 0.01416; approximate F = 3.94; p < 0.0001						
Variables	Wilk's lambda	Partial Wilks' lambda	F	Level p	Tolerance	R <sup>2</sup>
<i>Solidago canadensis</i> – root	0.0202	0.6988	0.79	0.5959	0.8510	0.14
<i>Solidago canadensis</i> – aerial part	0.0230	0.6113	1.16	0.3976	0.7658	0.32
<i>Solidago gigantea</i> – root	0.0807	0.1754	8.61	0.0012	0.9342	0.06
<i>Solidago gigantea</i> – aerial part	0.0862	0.1641	9.33	0.0008	0.6825	0.31

negatively affected the mass of *Ch. album* compared to the control treatment. Significant distances of Mahalanobis were recorded between 5% and 10% crude extract and 5% and 10% saponin fraction.. The first group is control, 5% saponin fraction and 5% crude extract. The second one-element group consists of a crude extract of 10%. These extracts are marked by a significant Euclidean distance in relation to the third group of 5% phenolic fraction, 10% saponin fraction and 10% phenolic fraction. The presented dendrogram determines the Euclidean distance between the tested extracts in the 4-dimensional space, but does not take into account the correlation between the variables studied. Therefore, some distances of Mahalanobis do not reflect the remoteness of individual clusters in the Euclid-

ean space. However, there is some convergence of results. Noteworthy is the small Euclidean distance between the control and the 5% saponin fraction, which is also confirmed by the non-significant distance of Mahalanobis (Figure 2).

*Papaver rhoeas* was characterized by decreased resistance to applied plant extracts (Table 7). No significant differences were found between the control and the types of extracts analyzed. Increased effectiveness in limiting the mass of this weed was demonstrated by 10 percent extracts and phenolic fractions or saponins obtained from *S. canadensis* root. The lack of significant variability between the tested extracts of the analyzed goldenrod species is confirmed by Wilks' lambda for total discrimination (Table 8). Also the squares of Mahalanobis distance

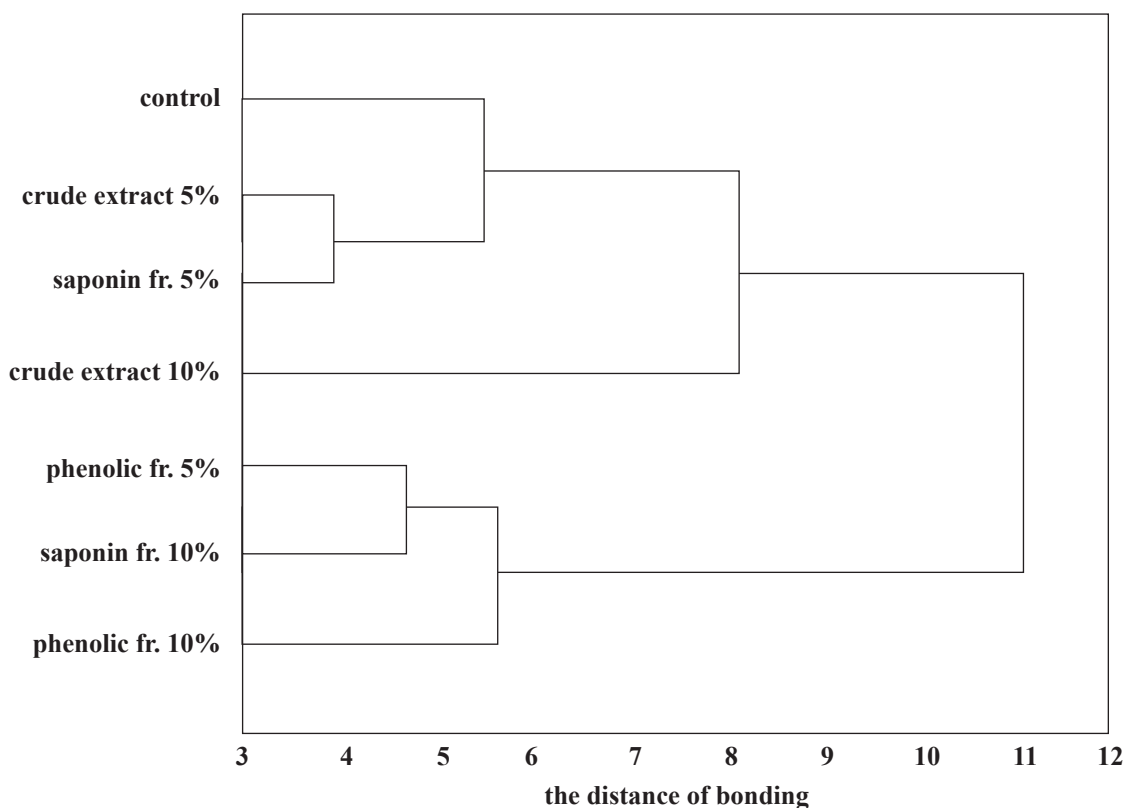


Figure 2. Dendrogram cluster analysis – dry mass of *Ch. album* under the conditions of various extracts.

Table 6. Squared Mahalanobis distances for *Chenopodium album* treated with individual fractions obtained from *Solidago*.

	control	crude extract 5%	crude extract 10%	phenolic fraction 5%	phenolic fraction 10%	saponin fraction 5%	saponin fraction 10%
control	0.000	26.610*	86.160**	18.787	33.818	14.652	68.092
crude extract 5%	26.610*	0.000	21.178*	19.502*	24.656*	2.105	39.291**
crude extract 10%	86.160**	21.178*	0.000	48.760**	35.389**	31.872**	32.300**
phenolic fraction 5%	18.787*	19.502*	48.760**	0.000	6.735	15.051	18.831*
phenolic fraction 10%	33.818**	24.656*	35.389**	6.735	0.000	21.245*	7.935
saponin fraction 5%	14.652	2.105	31.872**	15.051	21.245*	0.000	42.284**
saponin fraction 10%	68.092**	39.291**	32.300**	18.831*	7.935	42.284**	0.000

\*\* significant differences at the level  $p=0.01$

\* significant differences at the level  $p=0.05$

Table 7. Average dry matter of *Papaver rhoeas* depending on the applied extract from selected parts of plants of *Solidago gigantea* and *S. canadensis*.

Type of extract	<i>S. canadensis</i> root	<i>S. canadensis</i> aerial part	<i>S. gigantea</i> root	<i>S. gigantea</i> aerial part	Average
Control	0.57	0.57	0.57	0.57	0.570
Crude extract – 5%	0.57	0.57	0.57	0.63	0.585
Crude extract – 10%	0.37	0.43	0.50	0.60	0.475
Phenolic fraction – 5%	0.63	0.80	0.63	0.60	0.665
Phenolic fraction – 10%	0.33	0.63	0.47	0.43	0.465
Saponin fraction – 5%	0.43	0.77	0.70	0.73	0.658
Saponin fraction – 10%	0.33	0.57	0.57	0.53	0.500
Average	0.461	0.620	0.573	0.584	0.560

LSD *Solidago* species = 0.142; LSD types of extracts = not significant

LSD interaction (parts of plant of *Solidago* species)  $\times$  (types of extracts) = not significant

Table 8. Results of discriminant function analysis for *Papaver rhoeas*, treated with extracts from roots and aerial parts of *Solidago* species (*S. canadensis*, *S. gigantea*).

Wilks' lambda = 0.2371; F (24.39) = 0.8422; $p < 0.6673$						
Variables	Wilks' lambda	partial Wilks' lambda	F	Level p	Tolerance	R <sup>2</sup>
<i>Solidago canadensis</i> – root	0.4355	0.5446	1.5333	0.2549	0.7847	0.2153
<i>Solidago canadensis</i> – aerial part	0.2878	0.8240	0.3917	0.8695	0.9771	0.0229
<i>Solidago gigantea</i> – root	0.2855	0.8306	0.3738	0.8808	0.8668	0.1332
<i>Solidago gigantea</i> – aerial part	0.3294	0.7198	0.7135	0.6467	0.7915	0.2085



between the control and the extracts, phenolics or saponins fractions confirm the lack of effectiveness of plant extracts for this weed species (Table 9). The lack of effectiveness of analyzed extracts, regardless of the *Solidago* species, was also demonstrated in the case of *A. githago* (Table 10). The lambda Wilks partial values as well as the irrelevant Mahalanobis distances between the control and the tested extracts confirm the ineffectiveness of the analyzed extracts to reduce the weight of *A. githago* (Table 11). Multivariate analysis of variance MANOVA also showed negligible impact of the studied goldenrod species on the variability of the weight of this weed species under the influence of the investigated extracts, saponin fractions and phenolic fractions (Table 12).

The information available in the literature suggests that *Solidago* species can have a very strong impact on other plant species that grow in close proximity to each other. Most often it will be an inhibitory effect, although there have also been cases of stimulation of growth (Yang et al., 2007, Tang et al., 2009, Zhang et al., 2009). The effect of goldenrod on some species is due to the content of secondary metabolites in their cells, such as polyacetylates, diterpenoids, saponins, phenols or essential oils, which under favorable environmental conditions may exhibit allelochemical properties (Inose et al., 1991; Lu et al., 1993; Lu et al., 1995; Tori et al., 1999; Choi et al., 2004; Lendl, Reznicek, 2007). Judžentiene et al. (2023) showed that the leaf extract of *S. canadensis* caused a strong inhibitory effect on the test plants (*Lactuca sativa* and *Lepidium sativum*). In the studies of Pisula and Meiners (2010) it was shown that after the use of aqueous extract from *S. canadensis* and *S. gigantea* leaves, a strong inhibitory effect on the growth of *Raphanus sativus* was observed. Also, in the studies conducted by Kieć and Wiczorek (2009) regarding the suitability of various extracts and stocks for limiting the biomass of the *Ch. album*, most of them show an inhibitory effect. Similar results were achieved by Baličević et al. (2015). In their studies, aqueous extracts obtained from *S. gigantea* leaves inhibited the sprouting of *Amaranthus retroflexus* L., *Daucus carota* L., *Coriandrum sativum* L. and *Hordeum sativum* L. According to other studies, water extracts obtained from goldenrod also effectively limited germination and inhibited the initial development of wheat plants and *Matricaria maritima* L. ssp. *inodora* (L.) (Ravlić et al., 2015).

Secondary metabolites included in the roots, in addition to the effects on plants, also showed activity against fungi and cyanobacteria. Liu et al. (2016) showed that the essential oils obtained from *S. canadensis* effectively inhibited the growth of the fungus *Botrytis cinerea* Pers. infecting strawberries, also ensuring their better storage. Huang et al. (2013) demonstrated that alcohol extracts from the aerial parts of *S. canadensis* inhibited the growth of cyanobacteria from the *Microcystis aeruginosa* Kützinger species. Aqueous extracts of *Solidago canadensis* and *Solidago*

*gigantea* had no antifungal activity. However, antimicrobial activity was demonstrated, with root extracts having a stronger effect on bacteria than leaf extracts (Anžlovar, Koce, 2014). The antibacterial and antimutagenic activities of hexane and ethanol extracts from the aboveground parts of three goldenrod species (*Solidago virgaurea* L., *Solidago canadensis* L. and *Solidago gigantea* Ait.) were analysed. It was found that the most volatile compounds were present in the extracts obtained from *S. canadensis* and *S. gigantea*. In the former case, 62 compounds were detected in the ethanol extracts and 46 in the hexane extracts, while for *S. gigantea* it was 73 and 45 compounds, respectively. The tested extracts of these two goldenrod species were shown to exhibit antimicrobial activity, with strong activity against Gram-positive bacteria (*Staphylococcus aureus*, *Staphylococcus faecalis* and *Bacillus subtilis*) and slightly weaker activity against Gram-negative bacteria (*Escherichia coli*, *Klebsiella pneumoniae*, *Pseudomonas aeruginosa*). It was also shown that hexane extracts of *S. virgaurea*, *S. canadensis* and *S. gigantea* herb were characterized by antimutagenic activity, while ethanolic extracts showed no such effect (Kołodziej et al., 2011). There are also reports on the stimulation of the influence of extracts obtained from various goldenrod species on the development of other plants. However, in no case was such an intensive growth stimulation obtained, as after using the extracts tested by Domaradzki et al. (2017) with the addition of an adjuvant. *P. rhoeas* plants treated with *S. gigantea* extracts with the addition of an adjuvant containing fatty acid methyl esters of rapeseed oil had a mass increase of 3.82% to 8.18% compared to the control treatment.

Bing-Yao et al. (2006), using in their studies water and ethanolic extracts made from the rhizomes of *S. canadensis*, showed an increase in germination of seeds of *Brassica napus* L. var. *napus*. A study conducted by Baličević et al. (2015) shows that aqueous extracts obtained from *S. gigantea* leaves can stimulate the seedlings of *Abutilon theophrasti* Medik. for faster growth. In the studies carried out by Gruřová et al. (2016), methanol extracts from two goldenrod species (*S. canadensis* and *S. gigantea*) caused a significant positive effect on the elongation growth of *Raphanus sativa* L. and *Lepidium sativum* L. In laboratory tests, Affek-Starczewska and Rzymowska (2012) showed that the presence of goldenrod seeds in the vicinity of cereal grains significantly differentiated the length of roots and sprouts of three cereal species (wheat, barley and oats). Higher concentration of goldenrod seeds stimulated the growth of these plants, which could be evidence of allelopathic effects of the goldenrod. Gala-Czekaj et al. (2022) showed that aqueous extracts prepared from different plant parts of *Solidago canadensis* and *Solidago gigantea* are characterized by autoallelopathic growth-inhibiting properties of goldenrods at early stages of development. Furthermore, it has been experimentally shown that the content of phenolic compounds, antioxidants, selected phytohor-

Tabela 9. Squared Mahalanobis distances for *Papaver rhoeas* treated with individual fractions obtained from *Solidago*.

	control	crude extract 5%	crude extract 10%	phenolic fraction 5%	phenolic fraction 10%	saponin fraction 5%	saponin fraction 10%
control	0.000	0.402	3.720	1.492	3.578	8.007	3.851
crude extract 5%	0.402	0.000	2.907	1.709	4.416	6.010	3.750
crude extract 10%	3.720	2.907	0.000	7.850	2.501	6.196	1.222
phenolic fraction 5%	1.492	1.709	7.850	0.000	6.926	6.896	6.641
phenolic fraction 10%	3.578	4.416	2.501	6.926	0.000	9.862	1.552
saponin fraction 5%	8.007	6.010	6.196	6.896	9.862	0.000	4.620
saponin fraction 10%	3.851	3.750	1.222	6.641	1.552	4.620	0.000

\*\* significant differences at the level  $p=0.01$

\* significant differences at the level  $p=0.05$

Table 10. Average dry matter of *Agrostemma githago* depending on the applied extract from selected parts of plants of *Solidago canadensis* and *S. gigantea*.

Type of extract	<i>S. canadensis</i> root	<i>S. canadensis</i> aerial part	<i>S. gigantea</i> root	<i>S. gigantea</i> aerial part	Average
Control	8.60	8.60	8.60	8.60	8.60
Crude extract – 5%	9.33	8.83	9.07	9.80	9.26
Crude extract – 10%	8.23	8.57	8.73	8.67	8.55
Phenolic fraction – 5%	9.57	9.47	8.47	10.17	9.42
Phenolic fraction – 10%	9.37	9.33	7.97	8.67	8.84
Saponin fraction – 5%	9.37	9.67	9.63	8.70	9.34
Saponin fraction – 10%	9.23	7.73	8.93	8.57	8.62
Average	9.10	8.89	8.77	9.03	8.95

LSD *Solidago* species = not significant; LSD types of extracts = not significant

LSD interaction (parts of plant of *Solidago* species)  $\times$  (types of extracts) = not significant

Table 11. Results of discriminant function analysis for *Agrostemma githago*, treated with extracts from roots and aerial parts of *Solidago* species (*S. canadensis*, *S. gigantea*).

Wilks' lambda = 0.1572; F (24.39) = 1.1540; $p < 0.3366$						
Variables	Wilks' lambda	partial Wilks' lambda	F	Level p.	Tolerance	R <sup>2</sup>
<i>Solidago canadensis</i> – root	0.2713	0.5793	1.3314	0.3216	0.8065	0.1935
<i>Solidago canadensis</i> – aerial part	0.2356	0.6671	0.9150	0.5192	0.9320	0.0680
<i>Solidago gigantea</i> – root	0.2271	0.6921	0.8157	0.5796	0.7775	0.2225
<i>Solidago gigantea</i> – aerial part	0.2882	0.5454	1.5283	0.2563	0.9416	0.0584

Tabela 12. Squared Mahalanobis distances for *Agrostemma githago* treated with individual fractions obtained from *Solidago*.

	control	crude extract 5%	crude extract 10%	phenolic fraction 5%	phenolic fraction 10%	saponin fraction 5%	saponin fraction 10%
control	0.000	5.923	0.671	11.759	4.661	4.736	1.946
crude extract 5%	5.923	0.000	7.928	1.847	5.669	3.438	6.189
crude extract 10%	0.671	7.928	0.000	15.200	8.581	6.535	4.110
phenolic fraction 5%	11.759	1.847	15.200	0.000	6.338	6.989	12.048
phenolic fraction 10%	4.661	5.669	8.581	6.338	0.000	5.287	4.567
saponin fraction 5%	4.736	3.438	6.535	6.989	5.287	0.000	6.292
saponin fraction 10%	1.946	6.189	4.110	12.048	4.567	6.292	0.000

\*\* significant differences at the level  $p=0.01$

\* significant differences at the level  $p=0.05$

mones in the aqueous extracts varies between goldenrod species, as does their autotoxic effect. *Solidago canadensis* is more susceptible to autotoxin effects. Šmid et al. (2023) demonstrated the usefulness of *Solidago gigantea* extracts in combination with chitosan to develop eco-friendly textiles with antioxidant and UV-protective properties. Such textiles could be used as protective and therapeutic garments for people who are exposed to high doses of UV radiation on a daily basis and for long periods of time or who have skin problems and, due to their antioxidant properties, promote healing of various skin infections and diseases.

### CONCLUSIONS

1. The main components of the herb of both *Solidago* species are flavonoid glycosides, and phenolic acids which a high content of caffeoylquinic acid esters was characteristic of the above-ground parts of the plants.

2. The crude extract (5% w/v), phenolic fraction (5% w/v) and saponin fraction (10% w/v) from *S. gigantea* are characterized by high phytotoxicity towards *Ch. album*. Extracts from *S. canadensis* had an effect on *Ch. album* much weaker.

3. In the case of *P. rhoeas* treatment with *S. gigantea* extracts, the biomass reduction was 15% for the phenolic fraction and 35% for the saponin fraction. In the case of *S. canadensis* extracts, the allelopathic activity was not so pronounced and for the saponin fraction it ranged from 20 to 30%.

4. *Agrostemma githago* showed the highest resistance to preparations of both species. Extracts from *S. gigantea* had no effect on this species at all, while extracts from *S. canadensis* reduced biomass by a maximum of about 20%.

5. Extracts from the above-ground part of *S. gigantea* showed significantly higher herbicidal activity than extracts from the underground part and extracts from *S. canadensis*.

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