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ORIGINAL ARTICLE

Methanolic extracts of *Solidago gigantea* leaves as sustainable antifeedants against a stored pest: *Sitophilus granarius*

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Abstract

The development of natural insecticides is essential to minimize the adverse effects of synthetic plant protection products on the environment, human health, and non-target organisms. Natural bioactive compounds are considered safe, biodegradable, and often more selective, making them promising alternatives for sustainable pest management. Controlling insect pests in stored food products remains challenging due to restrictions on the use of chemical insecticides, which can leave harmful residues. Therefore, identification of plant-derived antifeedants is a viable solution. This study aimed to evaluate the antifeedant activity of methanolic leaf extracts from the invasive giant goldenrod (*Solidago gigantea* Aiton) against the grain weevil (*Sitophilus granarius* L.), a major storage pest. The chemical composition of the extracts was analyzed by LC-MS/MS, which identified 12 compounds. Chlorogenic acid, quercetin, and rutin were found at the highest concentrations, while citric acid, gallic acid, and kaempferol-3-rutinoside were present at the lowest concentrations. Antifeedant activity was assessed using the "wheat wafer test" at three extract concentrations (3.5, 5.0, and 12.0 mg · ml^{-1}) dissolved in ethanol. The extracts demonstrated medium to good antifeedant activity against female grain weevils and weak to medium effects against males. A significant reduction in feeding was observed in most cases, depending on both the sex of the insect and the extract concentration. Methanolic leaf extracts of *S. gigantea* exhibited promising antifeedant properties and may serve as potential natural insect deterrents. Further studies are required to evaluate their efficacy, safety, and mode of action under storage and field conditions.

Keywords: botanical pesticides, ecofriendly insecticide, feeding deterrence, integrated pest management, storage grain protection

Introduction

A wide range of agricultural commodities, including cereals, oilseeds, legumes, and fruits, undergo postharvest drying and storage to ensure year-round availability. Despite being classified as non-perishable, these products remain vulnerable to deterioration, particularly during storage and transportation. Global postharvest losses are estimated at 5–10%, reaching up to 30% in tropical climates and low-income regions (Pimentel 2011; Kumar and Kalita 2017; Saad and Abdelgaleil 2018). These losses result from both abiotic stressors, such as excessive heat and moisture, and biotic factors, primarily insect pests, along with other microorganisms and arthropods (Stejskal *et al.* 2015; Hagstrum and Phillips 2017; Hengsdijk and De Boer 2017; Hubert *et al.* 2018).

Insect pests inflict direct feeding damage and contaminate food with fecal matter, secretions, body fragments, or dead individuals. Additionally, mold proliferation and the associated production of mycotoxins exacerbate quantitative and qualitative losses, posing

significant health risks to humans and animals (Keskin and Ozkaya 2013; Stejskal *et al.* 2014). Collectively, these pest-induced damages contribute substantially to global food losses, which the Food and Agriculture Organization (FAO) estimates to exceed one billion tons annually (Gustavsson *et al.* 2013; Nayak and Daglish 2018). Storage insect pests are responsible for substantial damage to various crops, with losses ranging from 10–30%. In some cases, specific pests cause extreme damage, for example, *Leucinodes orbonalis* (Lepidoptera: Crambidae) causes 70–75% damage in brinjal, and *Helicoverpa armigera* (Lepidoptera: Noctuidae) causes 50–80% damage in tomatoes (Hasan *et al.* 2025). Overall, losses along storage and supply chains can reach 20–40%, with pest insects being a primary contributor to grain losses and a major factor in the global food crisis (Kostyukovsky *et al.* 2016).

Among stored-product pests, the grain weevil (*Sitophilus granarius* L., Coleoptera: Curculionidae) is one of the most economically significant, affecting cereals worldwide (Boniecki *et al.* 2020; Keszthelyi *et al.* 2021). Infestation by *S. granarius* progressively deteriorates grain quality, reducing hectoliter weight and total starch content, and altering protein composition during extended storage (Strelec *et al.* 2014). Infested grains also exhibit decreased kernel and test weights, lower fat content, and increased ash and protein levels (Keskin and Ozkaya 2015). Furthermore, *S. granarius* negatively affects seed viability by reducing germination rates and increasing moisture content in stored grains, which promotes fungal and bacterial growth (Keskin and Ozkaya 2015; Yousuf *et al.* 2025).

Various control methods have been explored, including residue-free approaches such as infrared irradiation (Keszthelyi *et al.* 2021) and automated visual detection systems for identifying damaged kernels and adult weevils in storage facilities (Boniecki *et al.* 2020). Despite these advances, managing grain weevils remains challenging due to their ability to survive extreme environmental conditions and reproduce rapidly at high temperatures (Athanassiou *et al.* 2019). Traditional chemical insecticides are increasingly constrained by stricter regulations and growing consumer demand for residue-free products (Niedermayer *et al.* 2016). In Poland, chemical control strategies rely on insecticides such as deltamethrin and pirimiphos-methyl, as well as fumigants such as aluminum phosphide (Olejarski and Węgorek 2013; Andrić *et al.* 2014; Quellhorst *et al.* 2023). While effective, these chemical plant protection products pose risks to human health and the environment, leading to a decline in approved substances and increasing interest in safer, plant-based alternatives that offer sustainability and minimal residue concerns (Kroos and Klementz 2016; Hasan *et al.* 2023; Uikey 2024).

In this context, invasive plant species have emerged as potential sources of environmentally friendly pest control agents. Several species, including *Ailanthus altissima* (Simaroubaceae), *Fallopia japonica* (Polygonaceae), *Solidago canadensis* (Asteraceae), and *Rhus typhina* (Anacardiaceae), contain bioactive compounds with insecticidal and herbicidal properties (Laznik *et al.* 2018; Chaw *et al.* 2019; Kozuharova *et al.* 2022). Among them, *Solidago* species have received particular attention for their bioactive properties. The essential oils of *S. canadensis* and *S. gigantea* exhibit insecticidal activity against a range of pests while demonstrating low toxicity to non-target organisms (Benelli *et al.* 2019). Moreover, extracts from these species have allelopathic effects against cereal weeds, with *S. gigantea* showing high phytotoxicity against *Chenopodium album* (Mołdoch and Domaradzki 2024). *Solidago* plants contain various bioactive compounds, including flavonoids, triterpene saponins, and terpenes, which contribute to pharmacological properties such as diuretic and antiseptic effects (Jiang 2006; Shelepova *et al*. 2021).

Solidago gigantea, commonly known as giant goldenrod, was first introduced in Poland in 1836 for cultivation and was recorded in the wild in 1853 in Lower Silesia (Guzikowa and Maycock 2014). Currently, it is widespread across Poland, particularly in the southern regions, and often predominates among local goldenrod species (Chmura *et al.* 2015). Morphologically, it is distinguished by glabrous shoots, compact panicles, and finely serrated or smooth leaf blades, in contrast to the pubescent shoots and spreading panicles of *S. canadensis* (Shelepova and Vinogradova 2021). *Solidago* species are highly invasive, forming dense stands that outcompete native vegetation and reduce biodiversity (Szymura and Szymura 2016; Szymura *et al.* 2018). Considering their allelopathic and insecticidal properties, invasive *Solidago* species are promising candidates for the development of natural pest control agents (Mołdoch and Domaradzki 2024; Benelli *et al.* 2019). However, it remains unclear whether leaf extracts of giant goldenrod influence the feeding behavior of economically important storage pests, such as S. granarius.

This study aimed to evaluate the effects of methanolic extracts of *S. gigantea* leaves on the feeding activity of the grain weevil (*S. granarius*). It was hypothesized that the extracts would inhibit feeding in a dose-dependent manner and that the effect might differ between male and female weevils. Feeding deterrent activity was assessed using *S. granarius*, a cosmopolitan and synanthropic pest commonly found in granaries, mills, warehouses, and households where grains are improperly stored. This species primarily feeds on wheat, rye, oats, barley, millet, buckwheat,

and maize, and has long served as a model organism for evaluating the deterrent effects of chemical compounds (Nawrot *et al.* 1986; Jackowski *et al.* 2015, 2017).

Materials and Methods

Collection of plant material

Aerial parts of giant goldenrods were collected in July 2024 from a fallow field situated in the northern region of Wrocław, Poland (51°09'46.8'' N, 17°06'53.9'' E). The plant material was harvested at the phenological stage when the leaves were fully developed but before the onset of flowering. The collected plant material was manually separated, and the leaves were isolated for further processing. The leaves were dried in the dark at temperatures not exceeding 50°C until a constant dry weight was achieved. The dried leaves prepared in this manner were used to prepare the methanolic extracts.

Preparation of methanolic extract from *Solidago gigantea* leaves

Giant goldenrod leaves were extracted according to the method described by Pachura *et al.* (2022), with minor modifications. In summary, dried and ground leaves (20 ± 0.5 g) were placed in a 250 ml glass flask and macerated with 200 ml of 80% analytical grade methanol for 24 h at 120 rpm. After extraction, the mixture was centrifuged for 30 minutes at 5,000 rpm, filtered, and evaporated to dryness. The resulting dry extract was weighed using an analytical balance. This extract was used for both antifeedant testing and chemical profiling.

For LC-MS/MS analysis, 10 mg of the extract was dissolved in 10 ml of high-purity chromatographic-grade methanol. Depending on the target analyte, the samples were diluted 1:100 (for hyperoside and chlorogenic acid) or 1:10 (for the remaining compounds) before the analysis. Prior to analysis, extracts were stored in a freezer to ensure their stability. All the chemicals and reagents used in this study were obtained from Sigma-Aldrich (Steinheim, Germany). All extractions and subsequent analyses were performed in triplicate.

Chemical analyses of the methanolic extract from *Solidago gigantea* leaves

LC-MS/MS analysis was carried out using an LC-MS-8045 system (Shimadzu, Kyoto, Japan) equipped with an Ac-Cucore™ RP-MS column (Thermo Fisher Scientific, Waltham, MA, USA) with specifications of 2.6 µm particle size, 100 Å pore size, and dimensions of 150 × 3.0 mm. The mobile phase consisted of 0.1% formic acid in water (eluent A) and 0.1% formic acid in acetonitrile (eluent B). A gradient elution program was applied as follows: starting at 10% B, increasing to 20% B for 5 min, then to 60% B for 10 min, followed by a return to 10% B for 13 min, which was then maintained for 17 min. The column flow rate was set at 0.35 ml · min^{-1}, and the oven temperature was maintained at 45°C. Table 1 presents details of the plant material analysis. Quantitative analysis was based on a five-point calibration curve of pure standards. The compounds for quantification were selected based on a qualitative screening analysis.

Table 1. Details of MRM analysis of compounds present in methanolic extracts of *Solidago gigantea* leaves

Compound	Ionization mode	Precursor ion [m/z]	Product ions [m/z]
Chlorogenic acid	negative	353.0	191.3 85.0 93.05
Citric acid	negative	137.4	92.95 65.05
Gallic acid	negative	169.4	124.95 79.80 69.00
Hyperoside	negative	463.3	300.1 271.15 301.1
Isorhamnetin--3-rutinoside	negative	623.2	315.15 314.10 299.15
Kempferol--3-rutinoside	negative	593.2	285.15 284.15 255.15
Luteolin--7-glucoside	positive	449.2	287.0 384.85 417.05
Quercetin	negative	301.2	151.0 179.0 121.0
Rutin	negative	609.3	300.15 301.1 271.25

Tests of feeding deterrent activity

For experimental purposes, *S. granarius* individuals were reared in constant darkness in climate-controlled chambers at 24 ± 1°C and 70 ± 5% relative humidity. Wheat grains of the NATULA variety were used as both a food source and an oviposition substrate.

Wheat wafer test

The wheat wafer disk bioassay is a standard method for evaluating the feeding deterrent activity of various compounds against insect pests (Aznar-Fernández *et al.* 2019; Larson *et al.* 2020; Zhang *et al.* 2020; Kerebba *et al.* 2022). This procedure was performed according to the protocol established by Nawrot *et al.* (1986), and later adapted by Jackowski *et al.* (2015, 2017). For bioassays, three concentrations of methanolic extract from giant goldenrod leaves were prepared: 3.5 mg · ml^{-1}, 5.0 mg · ml^{-1}, and 12 mg · ml^{-1}. These concentrations were selected based on preliminary tests to evaluate dose-dependent effects on feeding activity. The extracts were prepared using 99.9% ethanol as the solvent with a single drop (approximately 0.05 ml) of Tween 80 acting as an emulsifier.

Wheat wafer disks, 1.5 cm in diameter, were cut using a cork borer. They were immersed in the respective extract solutions or solvent (control) using forceps. The treated and control wafers were then placed on separate 210 mm diameter glass Petri dishes and air-dried for 30 minutes. After drying, the wafers were weighed and transferred to polystyrene Petri dishes with a diameter of 90 mm. Male or female *S. granarius* were introduced into Petri dishes containing wafers, which were sealed and placed under optimally controlled environmental conditions (24 ± 1°C, 70 ± 5% relative humidity). Adult grain weevils (at least 14 days old) were classified as male or female, based on morphological traits indicative of sexual dimorphism (Dinuță *et al.* 2009). Each treatment was replicated five times with five insects per replicate. After 120 h (five days), the insects were removed, and the remaining wafer material was weighed again to quantify the effect of the treatments.

Bioassays were performed under three experimental conditions: (1) reference treatment, in which both wafers were treated with the solvent only; (2) choice test, in which one wafer was treated with the extract and the other with the solvent; and (3) no-choice test, in which both wafers were treated with the extract. The amount of wafer material consumed was determined by comparing the initial and final weights of the wafers, which were used to calculate the relative (R), absolute (A), and total (T) deterrency coefficients.

Relative Deterrency Coefficient (R): This coefficient quantifies the difference in consumption between extract-treated and solvent-treated wafers in the choice test where both options are available. Formula:

$$R = \frac{C - E}{C + E} \times 100,$$

where:
C – amount of reference (solvent-treated) wafer consumed in the choice test;
E – amount of extract-treated wafer consumed in the choice test.

Absolute Deterrency Coefficient (A): This coefficient compares the consumption of wafers in the no-choice test (both extract-treated wafers) with the consumption in the reference test (both solvent-treated wafers). Formula:

$$R = \frac{CC - EE}{CC + EE} \times 100,$$

where:
CC – amount of reference (solvent-treated) wafers consumed in the no-choice test;
EE – amount of extract-treated wafers consumed in the no-choice test.

Total Deterrency Coefficient (T): The total deterrency coefficient is the sum of the relative and absolute deterrency coefficients. Formula:

$$T = A + R,$$

The T-coefficient is the primary measure for assessing the deterrent activity of a compound. T-values were interpreted as follows (Nawrot *et al.* 1986):

- Very Good Deterrent Activity: T-values between 151 and 200;
- Good Deterrent Activity: T-values between 101 and 150;
- Medium Deterrent Activity: T-values between 51 and 100;
- Weak Deterrent Activity: T-values below 50.

Negative T-values indicate the promotion of feeding rather than deterrence. It is important to note that R and A values should not be used independently to assess whether a compound deters or promotes feeding behavior.

For statistical analyses, in addition to the T-coefficient value, the weight loss of the wheat wafers from the choice test was used. Based on this weight loss, the inhibition of feeding caused by the giant goldenrod methanolic leaf extract was calculated using the following formula:

$$\text{Feeding inhibition} = \frac{ME - MC}{MC} \times 100\%,$$

where:
ME – amount of extract-treated wafer consumed in the choice test;
MC – amount of reference (solvent-treated) wafer consumed in the choice test.

Statistical analysis

The total deterrence coefficient (T) and wheat wafer mass loss (calculated using multiple-choice tests) were

used as indicators of the biological activity of the tested extracts. Data sets for each extract concentration and insect sex were tested for normality using the Shapiro-Wilk W test. As the data did not follow a normal distribution, nonparametric methods were applied. Kruskal-Wallis analysis of variance (ANOVA) by rank was used to compare the T-values obtained in the wafer tests at different concentrations of *S. gigantea* leaf extract for female and male insects. Pairwise comparisons of wheat wafer mass loss (extract-treated vs. solvent-only control) were performed using the Mann-Whitney U test, which was also applied to compare the T-coefficient values between male and female granary weevils. Statistical significance was set at $p \leq 0.05$. All analyses were performed using the STATISTICA software version 13 (TIBCO Software Inc., Palo Alto, CA, USA).

Results

Chemical profile of methanolic extracts from *Solidago gigantea* leaves

To isolate the non-volatile fraction of giant goldenrod, 80% [v/v] methanol was used, resulting in a total yield of 203 ± 5.1 mg per gram of dried material. Twelve compounds were identified in the plant extract. Among them, chlorogenic acid, quercetin, and rutin were quantified at the highest concentrations, whereas citric acid, gallic acid, and luteolin-7-glucoside presented the lowest concentrations. The detailed results are presented in Table 2. The main group of identified compounds was composed of flavonoids, whereas only a few phenolic acids and other organic acids were identified.

Table 2. Compounds identified in the methanolic extract from *Solidago gigantea* leaves

Compound	mg · 1 g^{-1} DM
3,4-dicaffeoylquinic acid	0.54 ± 0.03
3,5-dicaffeoylquinic acid	0.67 ± 0.03
Chlorogenic acid	5.21 ± 0.14
Citric acid	0.12 ± 0.01
Gallic acid	0.11 ± 0.01
Hyperoside	0.44 ± 0.03
Isorhamnetin-3-rutinoside	0.34 ± 0.05
Kaempferol	0.97 ± 0.01
Kaempferol-3-rutinoside	0.21 ± 0.01
Luteolin-7-glucoside	0.18 ± 0.02
Quercetin	2.10 ± 0.15
Rutin	2.51 ± 0.07

The effect of methanolic extracts from *Solidago gigantea* leaves on the inhibition of the feeding activity of *Sitophilus granarius*

Biotests conducted using the wheat wafer method allowed for the evaluation of the antifeedant properties of methanolic extracts of giant goldenrod leaves against the storage pest, *Sitophilus granarius*. The antifeedant activity of the extracts at different concentrations was assessed based on the total deterrency coefficient (T) (Fig. 1). The mean value of the T-coefficient was used to classify the intensity of the antifeedant effect. The extract at a concentration of 5.0 mg · ml^{-1} exhibited good feeding deterrent properties against female *S. granarius*, while the extracts at 3.5 mg · ml^{-1} and 12.0 mg · ml^{-1} showed medium deterrent effects (Fig. 1). Statistical analysis revealed significant differences in the values of the T-coefficient for females between concentrations of 3.5 mg · ml^{-1} and 5.0 mg · ml^{-1}

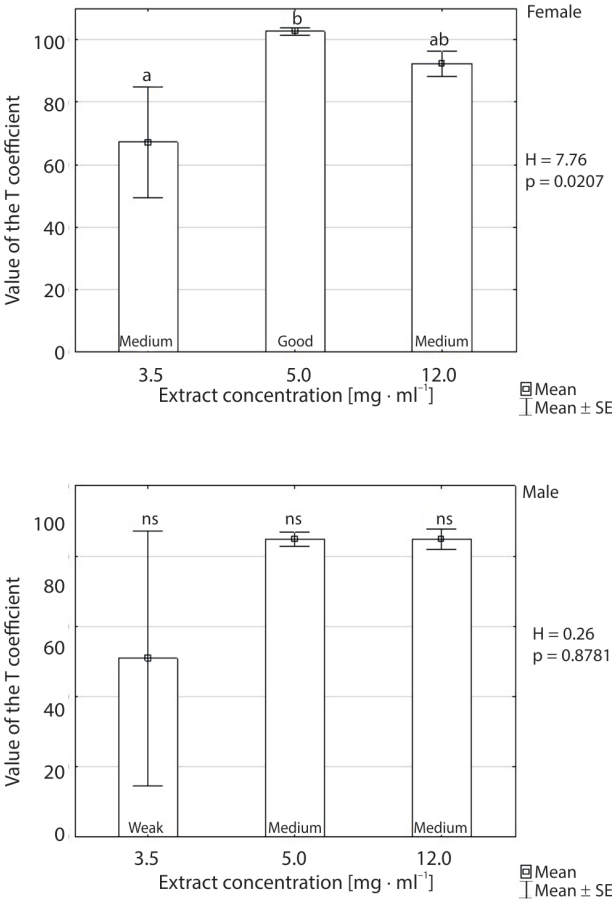

Fig. 1. Mean values of the total deterrency coefficient (T) as a result of exposure to different concentrations of the methanol extract of *Solidago gigantea* leaves. The results for *Sitophilus granarius* females are shown at the top and those for *S. granarius* males are shown at the bottom. Differences between concentrations were analyzed using the Kruskal-Wallis analysis of variance and lettered (H and p values are indicated on the right side of the graphs). Based on the average T-coefficient values, the deterrent activity was determined (from weak to very good).

(Fig. 1). In contrast, the inhibitory effect on male feeding activity was weak at 3.5 mg · ml^{-1} and medium at 5.0 mg · ml^{-1} and 12.0 mg · ml^{-1}. However, no statistically significant differences were observed in the T-coefficient values between the extract concentrations in male *S. granarius* (Fig. 1). A comparative analysis of the deterrency coefficient (T) between female and male grain weevils demonstrated that statistically significant sex-related differences were detected exclusively at the concentration of 5.0 mg · ml^{-1} of the methanolic extract from *S. gigantea* leaves, as confirmed by the Mann–Whitney U test (Table 3).

Table 3. Mean values ± standard error (SE) of the total deterrency coefficient (T) as a result of exposure to different concentrations of the methanol extract of *Solidago gigantea* leaves

Extract concentration [mg · ml^{-1}]	Female	Male	Z	p
	Mean ± SE			
3.5	67.12 ± 17.63	50.89 ± 36.35	0.20	0.83
5.0	102.6 ± 1.20	84.8 ± 1.99	2.50	0.01*
12.0	92.3 ± 4.04	84.9 ± 2.93	1.25	0.21

Z – standardized Mann–Whitney U test statistic; *p* – significance level; significant differences between sexes were determined using the Mann–Whitney U test and are marked with *, n = 5

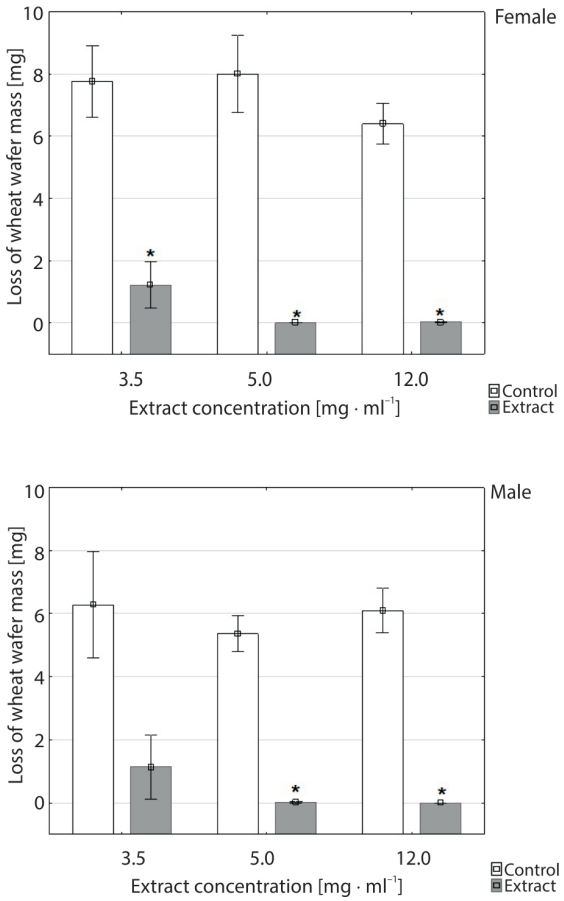

Fig. 2. Loss of wheat wafer mass and inhibition of feeding (%) in *Sitophilus granarius* females and males exposed to methanolic extracts of *Solidago gigantea* leaves at various concentrations (Mean ± SE). Significant differences between the control and each extracted concentration were determined using the Mann-Whitney U test and are marked with *, n = 5

The results for *Sitophilus granarius* females are shown at the top and those for *S. granarius* males are shown at the bottom. Differences between concentrations were analysed using the Kruskal-Wallis analysis of variance and lettered (H and p values are indicated on the right side of the graphs). Based on the average T-coefficient values, the deterrent activity was determined (from weak to very good) Table 3.

Further analyses were performed on the mass loss of wheat wafers in the choice test. It was found that, regardless of the extract concentration, the female grain weevils consumed significantly more control wafer disks than those treated with methanolic extracts from *S. gigantea* leaves, as determined by the Mann–Whitney U test (Fig. 2, Tab. 4). Furthermore, the inhibition of feeding of female grain weevils induced by the applied extracts was highest at a concentration of 5.0 mg · ml^{-1} (–100%), while at 3.5 mg · ml^{-1}, the inhibition was –84.3% (Fig. 2, Tab. 4). Male grain weevils consumed significantly more control wheat wafer disks than those treated with *S. gigantea* leaves extract when extracts with concentrations of 5.0 mg · ml^{-1} and 12.0 mg · ml^{-1} were applied (Fig. 2, Tab. 4). The strongest feeding inhibition in male grain weevils was observed at a concentration of 12.0 mg · ml^{-1} of the extract (–100%), while the weakest inhibition occurred at 3.5 mg · ml^{-1} (–81.8%) (Fig. 2, Tab. 4).

Table 4. Results of paired comparisons using the Mann–Whitney U test: Evaluation of the impact of *Solidago gigantea* leaf extract on the reduction in wheat wafer mass (control vs. treatment *S. gigantea* leaf extract) after 120 h of *S. granarius* feeding

Extract concentration [mg · ml^{-1}]	Female		Male	
	Z	p	Z	p
3.5	2.54	0.01*	1.88	0.59
5.0	2.67	0.01*	2.59	0.01*
12.0	2.59	0.01*	2.67	0.01*

Z – standardized Mann–Whitney U test statistic; *p* – significance level; statistically significant differences are denoted by asterisks (*), n = 5

Discussion

Plant-based substances are considered promising alternatives to synthetic insecticides for pest control, particularly in stored products and organic farming (Hasan *et al.* 2023). Plants produce diverse chemical

defence compounds that can effectively reduce insect feeding through multiple mechanisms, offering sustainable options for pest management. These natural substances include terpenes, flavonoids, alkaloids, polyphenols, and other secondary metabolites that act as antifeedants, repellents, and growth regulators (Souto *et al.* 2021). Plant defences operate through both direct mechanisms, affecting pest feeding behavior and survival, and indirect mechanisms, such as the release of volatile organic compounds that attract natural predators (Navya *et al.* 2025). Many of these compounds are constitutively present or induced upon herbivore attack, with plants capable of synthesizing over 25,000 distinct terpenoid structures (Navya *et al.* 2025). Particularly effective antifeedant sources include *Angelica archangelica* (Apiaceae), *Caesalpinia bonduc* (Caesalpiniaceae), *Grindelia camporum* (Asteraceae), and *Piper hispidinervum* (Piperaceae) (Pavela *et al.* 2025). Botanical pesticides, derived from plant extracts and essential oils, can repel, deter, or kill insect pests while being biodegradable, environmentally friendly, and posing minimal risks to human health compared with synthetic alternatives (Yactayo-Chang *et al.* 2020; Souto *et al.* 2021; Hasan *et al.* 2023). Nevertheless, despite their potential, challenges remain in the formulation, stability, and standardization of botanical pesticides. Their successful integration into pest management strategies could substantially contribute to more sustainable agricultural practices (Hasan *et al.* 2023).

Twelve different compounds were identified and quantified in the methanolic extracts of giant goldenrod. The number of compounds found corresponds to previous studies (Zekič *et al.* 2020; Radušienė *et al.* 2024). In some studies the number of compounds identified was higher; however, these studies were focused on other parts of the plant (such as roots) or used different extraction methods (Woźniak *et al.* 2018). Additionally, the extraction yield obtained in the present study (103 mg) and the concentration of particular compounds were different from those reported in earlier studies (Woźniak *et al.* 2018; Zekič *et al.* 2020), although the differences remained within the same order of magnitude. The observed differences may have occurred because the plant samples were collected at different times and locations and grown under different environmental conditions.

Recent studies have revealed the bioactive properties of giant goldenrod extract. Root extracts contain clerodane diterpenes with antibacterial and antifungal activities against various microorganisms (Móricz *et al.* 2021). Leaf extracts also contain antibacterial compounds, including solidagoic acids H, E, I, and F, which show moderate activity against gram-positive bacteria (Baglyas *et al.* 2022). High-performance thin-layer chromatography combined with bioautography and chemometrics has enabled the distinction of *Solidago* species based on their bioactive profiles, with solidagoic acid A and dialdehyde clerodane diterpene identified as the main bioactive markers of *S. gigantea* (Móricz *et al.* 2021). Furthermore, commercially available extracts of giant goldenrod exhibit notable bioactive properties. Root-derived clerodane diterpenes display antimicrobial activity against bacteria and fungi, as well as inhibitory effects on key enzymes such as acetyl- and butyrylcholinesterases, α- and β-glucosidases, and α-amylase (Móricz *et al.* 2021). In addition, *S. gigantea* extracts demonstrate strong antioxidant activity, with fractions showing IC_{50} values of 13.32–16.89 $\mu g \cdot ml^{-1}$ for lipid peroxidation inhibition (Woźniak *et al.* 2018). These findings highlight the diverse bioactive properties of *S. gigantea* and their potential applications.

Among the compounds identified in the methanolic extract of *S. gigantea* leaves, chlorogenic acid, quercetin, and rutin were present at the highest concentrations. Chlorogenic acid (CGA) has emerged as a potent plant defense compound against insect herbivores. Studies have demonstrated its efficacy in deterring insect growth and feeding across various species (Kundu and Vadassery 2019). CGA exhibits high toxicity and significant sublethal effects on *Bemisia tabaci* by prolonging developmental stages and reducing fecundity (Wang *et al.* 2023). Similar effects have been observed in *Mythimna separata*, in which CGA treatment extended larval instars and decreased survival rates (Lin *et al.* 2022). The insecticidal properties of CGA are related to its induced biosynthesis in plants after herbivore attack, as observed in sweet potato leaves that respond to weevil feeding (Liao *et al.* 2020). Insect detoxification mechanisms against CGA involve increased activity of P450 enzymes and upregulation of CYP450 genes (Lin *et al.* 2022; Wang *et al.* 2023). These findings highlight CGA's potential as a botanical insecticide. However, the effectiveness of CGA as a defense mechanism appears to be species-specific. Although it has shown promise against some insect herbivores (Kundu and Vadassery 2019), it did not demonstrate significant deterrence or toxicity against the alfalfa weevil *Hypera brunneipennis* in short-term behavioral assays and long-term fertilization tests (Bernays and Cornelius 1992).

Research on the role of quercetin as a deterrent against weevil beetles has yielded mixed results. Quercetin-3-O-rutinoside presented insecticidal activity against *Rhynchophorus ferrugineus* (Darrag *et al.* 2022). A comprehensive review by Riddick (2021) indicated that quercetin is generally nonharmful to Coleoptera, suggesting limited deterrent activity against beetles as a group. However, responses vary considerably between species. For instance, Riddick *et al.* (2018) reported that quercetin stimulated

oviposition in the ladybird beetle *Coleomegilla maculata*, with females preferentially laying eggs near quercetin sources. These findings underscore the complex and species-specific nature of quercetin–beetle interactions. However, quercitrin, a glycoside of quercetin, has demonstrated feeding deterrent effects in cotton square and plate bioassays (Bird and Hedin 1986). Quercetin isolated from the bark of *Bobgunnia madagascariensis* stems exhibited a feeding deterrent activity of more than 50% against the confused flour beetle (*Tribolium castaneum*) in a wafer disk choice bioassay (Adeyemi *et al.* 2010). Interestingly, when tested in Colorado potato beetle larvae (*Leptinotarsa decemlineata*), quercetin and other flavonoids were found to be almost ineffective in deterring feeding or inhibiting growth, whereas phenols and phenolic acids were highly efficient (Pavela 2007).

Another key flavonoid identified was rutin, which also demonstrated significant antifeedant and insecticidal effects. In *Helicoverpa armigera* and *Spodoptera litura*, rutin inhibits larval feeding, prolongs developmental time, and increases mortality (Jadhav *et al.* 2012). Similarly, in *Spodoptera frugiperda*, rutin extends larval development, reduces larval and pupal weights, and decreases pupal viability (Silva *et al.* 2016). These effects suggest the potential of integrated pest management strategies. However, their effectiveness varies among the species. Although it adversely affected lepidopteran pests, it had little or no effect on the Colorado potato beetle *Leptinotarsa decemlineata* (Pavela 2007). Interestingly, in the boll weevil *Anthonomus grandis*, rutin acted as a feeding stimulant in a plate bioassay (Bird and Hedin 1986).

The results of these biotests using the wheat wafer test method indicated that methanolic extracts from *S. gigantea* leaves exhibited antifeedant properties against *S. granarius*, and their effectiveness depended on the concentration of the extract and the sex of the insect. The deterrent effect was generally stronger in females than in males. Based on the total deterrency coefficient (T) values, the extracts exhibited medium to good feeding deterrent properties against female grain weevils, whereas their effects on males ranged from weak to medium. In the choice test, grain weevils consumed more control wheat wafers than those treated with methanolic extract from giant goldenrod leaves. For females, significant differences were observed at all tested extract concentrations, whereas for males, no statistically significant differences were detected at any of the concentrations. These findings suggest that *S. gigantea* extracts have the potential to treat *S. granarius* by effectively targeting female feeding behavior. Research has indicated that the effectiveness of insecticides on storage pests can vary depending on their sex. Studies have shown significant differences in susceptibility between male and female insects of various species. For example, male moths of three tortricid species are less susceptible to the organophosphate chlorpyrifos than females (Navarro-Roldán *et al.* 2017). In contrast, females are generally more susceptible to various grain protectants than males (Athanassiou *et al.* 2019). Sex-dependent effects were also observed with biosynthesized nanoparticles, where selenium nanoparticles decreased longevity in female *Callosobruchus maculatus* but extended it in males (San *et al.* 2023). These findings highlight the importance of considering sex ratios when evaluating insecticidal effectiveness (Fisher *et el.* 2024). The variability in susceptibility between sexes can be influenced by factors such as mode of action, target species, and even the presence of endosymbionts such as *Wolbachia* (San *et al.* 2023).

Research conducted by Herrera-Mayorga *et al.* (2022) demonstrated that another species of the genus *Solidago*, *S. graminifolia*, has insecticidal properties. The *S. graminifolia* extracts exhibited significant insecticidal activity against *Spodoptera frugiperda* larvae, and the ethanolic extract caused 81% mortality. Quercetin, a flavonoid found in *S. graminifolia*, has shown potent insecticidal activity. According to the authors, the toxic effect of the extracts depends on the solvent used for extraction, as the use of other organic solvents such as dichloromethane and hexane resulted in a weaker toxic effect on *S. frugiperda* larvae. Studies by other authors indicate that the essential oils of giant goldenrods and Canadian goldenrods can serve as sources of natural pesticides (Benelli *et al.* 2019). The toxic effects of these oils vary depending on the plant part used for extraction and the target pest species. The essential oil from the leaves of *S. canadensis* was only effective against the pests *Culex quinquefasciatus*, *S. littoralis*, and *Musca domestica* (Benelli *et al.* 2019). Essential oils from the leaves and flowers of *S. gigantea* exhibited the strongest activity against *S. littoralis*. Therefore, extracts from the leaves or other parts of giant goldenrods may exhibit stronger feeding deterrent activity against other storage pests. The effectiveness of the deterrent effect may also depend on the solvent used for extraction. Overall, these results support the potential of *S. gigantea* extracts, particularly leaves, as promising candidates for botanical insecticides to protect stored products.

Conclusions

In conclusion, the methanolic extracts of *S. gigantea* leaves exhibited antifeedant properties against the storage pest *S. granarius*. The effect of extracts on pest feeding depended on both the concentration used and the sex of the insect. Further research is necessary

to determine the insecticidal potential of *S. gigantea* against other insect pest species and elucidate its mode of action.

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