Analysis of the Essential Oil of *Dipsacus japonicus* Flowering Aerial Parts and its Insecticidal Activity against *Sitophilus zeamais* and *Tribolium castaneum*

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Water-distilled essential oil from the aerial parts of *Dipsacus japonicus* Miq. (Dipsacaceae) at the flowering stage was analysed by gas chromatography-mass spectrometry (GC-MS). Forty-six compounds, accounting for 96.76% of the total oil, were identified and the main compounds of the essential oil were linalool (11.78%), *trans*-geraniol (8.58%), 1,8-cineole (7.91%), β -caryophyllene (5.58%), α -terpineol (5.32%), β -selinene (5.15%), and spathulenol (5.04%). The essential oil of *D. japonicus* possessed contact toxicity against two grain storage insects, *Sitophilus zeamais* and *Tribolium castaneum* adults, with LD₅₀ values of 18.32 μ g/adult and 13.45 μ g/adult, respectively. The essential oil of *D. japonicus* also exhibited pronounced fumigant toxicity against *S. zeamais* (LC₅₀ = 10.11 mg/l air) and *T. castaneum* adults (LC₅₀ = 5.26 mg/l air). Of the three major compounds, 1,8-cineole exhibited stronger fumigant toxicity than the crude essential oil against *S. zeamais* and *T. castaneum* adults with LC₅₀ values of 2.96 mg/l air and 4.86 mg/l air, respectively.

Key words: *Dipsacus japonicus*, Essential Oil, Insecticidal Activity

Introduction

Botanical pesticides have the advantage of providing novel modes of action against insects that can reduce the risk of cross-resistance as well as offering new leads for the design of target-specific molecules. During a screening program for new agrochemicals from Chinese medicinal herbs and local wild plants, the essential oil of *Dipsacus japonicus* Miq. (family: Dipsacaceae) flowering aerial parts was found to possess insecticidal activity against the maize weevil (*Sitophilus zeamais* Motsch.) and the red flour beetle (*Tribolium castaneum* Herbst). *S. zeamais* and *T. castaneum* are two serious pests of stored grains worldwide (Liu and Ho, 1999). Infestations not only cause significant losses due to the consumption of grains, they also result in elevated temperature and moisture conditions that lead to an accelerated growth of molds, including toxic species (Magan *et al.*, 2003). Currently, control of stored-product insects relies heavily on the use of synthetic insecticides and

fumigants. However, repeated use of synthetic fumigants for decades has led to resurgence of stored-product insect pests, sometimes resulting in the development of resistance, and had undesirable effects on non-target organisms (Zettler and Arthur, 2000). These problems have highlighted the need to develop new types of selective insect control alternatives with fumigant action. Plant essential oils and their components have been shown to possess potential to be developed as new fumigants, and they may have the advantage over conventional fumigants in terms of low mammalian toxicity, rapid degradation, and local availability (Isman, 2006; Rajendran and Srianjini, 2008).

D. japonicus is a perennial herb, growing up to 1.5 m, and is mainly distributed in Central and Northern China as well as Japan and Korea (Flora of China Editorial Board, 1986). Dried roots of *D. japonicus* were used as Chinese medicinal herb, *Dipsaci Radix*, in the past (Jiangsu

New Medical College, 1977). But now, tap roots of *D. asperoides* are exclusively used as medicine (*Dipsaci Radix*), and *D. asperoides* is regarded an adulterant (Chinese Pharmacopoeia Commission, 2010). Several studies on chemical constituents of *D. japonicus* have been reported, and several saponins, triterpenoid glycosides, iridoid and bis-iridoid glycosides have been isolated (Wei *et al.*, 1995, 1998; Trinh *et al.*, 1999, 2002; Miao *et al.*, 2000a, b). However, a literature survey has shown that there is no report on the chemical composition of the essential oil derived from *D. japonicus*, and the insecticidal activity of this oil against stored-product insects has not been determined. The present investigation consisted of two parts: determination of the chemical composition of the essential oil of *D. japonicus* flowering aerial parts and evaluation of the essential oil as insecticide/fumigant for the control of the stored-product insect pests.

Material and Methods

Plant material

The flowering aerial parts of *D. japonicus* were collected in August 2009 from Xiaolongmen National Forest Park (39.48° N, 115.25° E), Mentougou District, Beijing, China. The samples were air-dried and identified by Dr. Q. R. Liu (College of Life Sciences, Beijing Normal University, Beijing, China). A voucher specimen (ENTCAU-Dipsacaceae-10002) was deposited at the Department of Entomology, China Agricultural University, Beijing, China. The samples were ground to a powder using a grinding mill (Retsch, Haan, Germany). A 600-g portion of powder was dispersed in 1,800 ml of distilled water and soaked for 3 h. The mixture was then boiled in a round-bottom flask and steam-distilled for 6–8 h. Separation of the essential oil from the aqueous layer was done in a separatory funnel, using the non-polar solvent *n*-hexane. The solvent was evaporated using a vacuum rotary evaporator (Büchi rotavapor R-124; Flawil, Switzerland). The resulting sample was dried over anhydrous Na₂SO₄ and kept at 4 °C for subsequent experiments. Linalool (98%), 1,8-cineole (98%), and *trans*-geraniol (98%) were purchased from Sigma-Aldrich Chemical Co. (St. Louis, MO, USA).

Insects

Maize weevils (*S. zeamais*) and red flour beetles (*T. castaneum*) were obtained from laboratory cultures maintained in the dark in incubators at 29–30 °C and 70–80% relative humidity. The red flour beetles were reared on wheat flour mixed with yeast (10:1, w/w), while maize weevils were reared on whole wheat grains at 12–13% moisture content in glass jars (diameter, 85 mm; height, 130 mm). About one-week-old, unsexed adult weevils/beetles were used in all experiments. All containers housing insects and the Petri dishes used in experiments were made escape-proof with a coating of polytetrafluoroethylene (Fluon, Blades Biological, Edenbridge, UK).

Gas chromatography-mass spectrometry (GC-MS)

The essential oil of *D. japonicus* was subjected to GC-MS analysis on an Agilent (Santa Clara, CA, USA) system consisting of a model 6890N gas chromatograph, a model 5973N mass selective detector (EIMS; electron energy, 70 eV), and an Agilent ChemStation data system. The GC column was an HP-5ms (Hewlett Packard, Santa Clara, CA, USA) fused silica capillary with a 5% phenyl-methylpolysiloxane stationary phase, a film thickness of 0.25 µm, a length of 30 m, and an internal diameter of 0.25 mm. The GC settings were as follows: The initial oven temperature was held at 60 °C for 1 min and ramped at 10 °C/min to 180 °C, held for 1 min, and then ramped at 20 °C/min to 280 °C and held for 15 min. The injector temperature was maintained at 270 °C. The sample (1 µl) was injected neat, with a split ratio of 1:10. The carrier gas was helium at a flow rate of 1.0 ml/min. Spectra were scanned from *m/z* 20 to 550 at 2 scans/s. Most constituents were identified by gas chromatography by comparison of their retention indices with those found in the literature or with those of authentic compounds available in our laboratories. The retention indices were determined in relation to a homologous series of *n*-alkanes (C₈–C₂₄) under the same operating conditions. Further identification was made by comparison of their mass spectra with those stored in NIST 08 and Wiley 275 libraries or with mass spectra available in the literature (Adams, 2007). Component relative percentages were calculated based on the normalization method without using correction factors.

Contact toxicity by topical application

The contact toxicity of the essential oil of *D. japonicus* and its major constituents against *S. zeamais* and *T. castaneum* adults was measured as described by Liu and Ho (1999). Range-finding studies were run to determine the appropriate testing concentrations of the essential oil/compounds. A serial dilution of the essential oil/compounds (6 concentrations) was prepared in *n*-hexane. Aliquots of 0.5 μ l/insect were topically applied dorsally to the thorax of the insects, using a Burkard Arnold microapplicator (Burkard Scientific Supply, Rickmansworth, England). Controls were determined using 0.5 μ l *n*-hexane/insect. Ten insects were used for each concentration and control, and the experiment was replicated six times. Both the treated and control insects were then transferred into glass vials (10 insects/vial) containing culture medium and kept in incubators at 29–30 °C and 70–80% relative humidity. Mortality of insects was observed after 24 h. The observed mortality data were corrected for control mortality using Abbott's formula. Results from all replicates were subjected to probit analysis using the PriProbit Program V1.6.3 to determine LD₅₀ values (Sakuma, 1998).

Fumigant toxicity bioassay

Range-finding studies were run to determine the appropriate testing concentrations of *D. japonicus* essential oil and its constituents. A Whatman filter paper (diameter, 2.0 cm) was placed on the underside of the screw cap of a glass vial (diameter, 2.5 cm; height, 5.5 cm; volume, 24 ml). Ten μ l of the essential oil/compounds (6 concentrations) were added to the filter paper. The solvent was allowed to evaporate for 15 s before the cap was placed tightly on the glass vial (with 10 unsexed insects) to form a sealed chamber. The vials were incubated at 27–29 °C and 70–80% relative humidity for 24 h. Mortality of insects was observed and corrected for control mortality using Abbott's formula. The results from all replicates were subjected to probit analysis using the PriProbit Program V1.6.3 to determine LC₅₀ values (Sakuma, 1998).

Results and Discussions

Essential oil analysis

The yield of the yellow essential oil from *D. japonicus* flowering aerial parts was 0.12% (v/w), and the density of the concentrated essential oil was 0.83 g/ml. A total of 46 compounds of the essential oil were identified, accounting to 96.76% of the total oil. The principal compounds in the essential oil were linalool (11.78%), *trans*-geraniol (8.58%), 1,8-cineole (7.91%), β -caryophyllene (5.58%), α -terpineol (5.32%), β -selinene (5.15%), and spathulenol (5.04%) (Table I). Monoterpenoids represented 21 of the 46 compounds, corresponding to 58.70% of the whole oil, while 17 of the 46 constituents were sesquiterpenoids (34.35% of the crude essential oil).

Insecticidal activity

The essential oil of *D. japonicus* flowering aerial parts possessed contact toxicity against *S. zeamais* and *T. castaneum* adults with LD₅₀ values of 18.32 μ g/adult and 13.45 μ g/adult, respectively (Table II). Compared with the positive control, pyrethrum extract (25% pyrethrin I and pyrethrin II), the essential oil of *D. japonicus* was 4 and 37 times less acutely toxic against the two species of grain storage insects. The pyrethrum extract had an acute toxicity to *S. zeamais* and *T. castaneum* with LD₅₀ values of 4.29 μ g/adult and 0.36 μ g/adult, respectively (Liu *et al.*, 2010).

The essential oil of *D. japonicus* also exhibited stronger fumigant toxicity against *T. castaneum* adults (LC₅₀ = 5.26 mg/l air) than *S. zeamais* adults (LC₅₀ = 10.11 mg/l air) (Table III). The commercial grain fumigant methyl bromide (MeBr) was reported to have fumigant activity against *S. zeamais* and *T. castaneum* adults with LC₅₀ values of 0.67 mg/l air and 1.75 mg/l air, respectively (Liu and Ho, 1999), thus the essential oil of *D. japonicus* was 15 and 3 times less toxic to *S. zeamais* and *T. castaneum*, respectively. However, considering that the commercial fumigants are synthetic insecticides, the fumigant activity of the essential oil of *D. japonicus* flowering aerial parts is quite promising. Moreover, compared with the other essential oils in previous studies, the essential oil of *D. japonicus* flowering aerial parts exhibited stronger or the same level of fumigant toxicity against the maize weevils, *e.g.* essential oils of *Artemisia lavandulaefolia* (LC₅₀ =

Table I. Chemical constituents of the essential oil derived from *Dipsacus japonicus* flowering aerial parts.

Peak no.	Compound	RI*	Content (%)
1	α -Pinene	931	3.57
2	β -Pinene	974	0.28
3	β -Myrcene	991	0.91
4	δ -3-Carene	1007	1.06
5	α -Terpinene	1017	0.67
6	p -Cymene	1025	0.34
7	1,8-Cineole	1032	7.91
8	γ -Terpinene	1059	0.78
9	Terpinolen	1089	0.56
10	Linalool	1094	11.78
11	Phenylethyl alcohol	1116	1.11
12	Camphor	1146	0.45
13	Borneol	1174	1.21
14	4-Terpineol	1179	2.93
15	p -Cymen-8-ol	1182	0.96
16	α -Terpineol	1189	5.32
17	Carvone	1238	1.38
18	<i>trans</i> -Geraniol	1252	8.58
19	Linalool acetate	1254	1.28
20	Bornyl acetate	1287	1.66
21	Thymol	1292	0.69
22	4-Vinylguaicol	1311	0.25
23	α -Cubebene	1350	2.66
24	Citronellol acetate	1354	0.01
25	Eugenol	1356	0.82
26	α -Copaene	1375	1.02
27	β -Elemene	1394	2.39
28	α -Gurjunene	1406	0.78
29	β -Caryophyllene	1420	5.58
30	<i>trans</i> - α -Ionone	1426	0.47
31	Dihydro- β -ionone	1433	1.49
32	(<i>Z</i>)- β -Farnesene	1438	0.43
33	Geranyl acetone	1458	3.88
34	γ -Selinene	1470	0.99
35	γ -Gurjunene	1473	1.26
36	β -Selinene	1489	5.15
37	γ -Cadinene	1513	1.56
38	Dihydroactinolide	1525	0.57
39	α -Calacorene	1546	0.76
40	Ledol	1562	0.69
41	Spathulenol	1578	5.04
42	Globulol	1582	0.01
43	Caryophyllene oxide	1583	2.79
44	β -Eudesmol	1648	2.43
45	α -Cadinol	1654	0.17
46	β -Bisabolol	1673	0.65
Total			96.76
Monoterpenoids			58.70
Sesquiterpenoids			34.35
Others			4.71

* RI, retention index as determined on an HP-5ms column using a homologous series of *n*-alkanes.

11.2 mg/l air; Liu *et al.*, 2010), *A. vestita* (LC₅₀ = 13.42 mg/l air; Chu *et al.*, 2010a), *Illicium simon-sii* (LC₅₀ = 14.95 mg/l air; Chu *et al.*, 2010b), *A. sieversiana* (LC₅₀ = 15.0 mg/l air; Liu *et al.*, 2010), and *Kadsura heteroclita* (LC₅₀ = 14.01 mg/l air; Li *et al.*, 2011). Compared with the other essential oils in the literature, the essential oil of *D. japonicus* possessed stronger fumigant toxicity against *T. castaneum* adults, e.g. essential oils of *Citrus reticulata* (LC₅₀ = 19.47 μ l/l air), *Schinus terebinthifolius* (LC₅₀ = 20.50 μ l/l air; Mohamed and Abdelgaleil, 2008), *Perovskia abrotanoides* (LC₅₀ = 11.39 μ l/l air; Arabi *et al.*, 2008), and *Drimys winteri* (LC₅₀ = 9.0–10.5 μ l/l air; Zapata and Smaghe, 2010), but was lesser toxic than the essential oil of *Laurelia sempervirens* (LC₅₀ = 1.6–1.7 μ l/l air; Zapata and Smaghe, 2010).

The three main constituents of *D. japonicus* essential oil, viz. 1,8-cineole, *trans*-geraniol, and linalool, exhibited contact toxicity against the two grain storage insects (16.34 μ g/adult, 15.11 μ g/adult, and 24.88 μ g/adult for *S. zeamais*, respectively, and 12.67 mg/adult, 21.34 mg/adult, and 8.12 mg/adult for *T. castaneum*, respectively) (Table II). Among the three compounds, 1,8-cineole possessed strongest fumigant toxicity against *S. zeamais* and *T. castaneum* adults with LC₅₀ values of 2.96 mg/l air and 4.86 mg/l air, respectively, while the two other compounds were as active or less active than the crude essential oil. It is suggested that the fumigant toxicity of the essential oil may mainly be attributed to 1,8-cineole, because it showed stronger fumigant toxicity than the crude essential oil (no overlap in 95% confidence limits) (Table II). Moreover, in previous studies, 1,8-cineole was found to exhibit fumigant toxicity against *T. castaneum* adults with LC₅₀ values of 41 μ l/l air (Suthisut *et al.*, 2011), 15.3 μ l/l air (Lee *et al.*, 2004), and 1.52 mg/l air (Tripathi *et al.*, 2001), respectively. 1,8-Cineole also possessed fumigant toxicity against *S. zeamais* adults with an LC₅₀ value of 2.96 mg/l air (Liu and Du, 2011). It also exhibited fumigant toxicity against several other stored-product insects and cockroaches as well as mosquitoes, e.g. the rice weevil (*S. oryzae*; LC₅₀ = 22.8 μ l/l air) and the lesser grain borer (*Rhyzopertha dominica*; LC₅₀ = 9.5 μ l/l air) (Lee *et al.*, 2004).

The above findings suggest that the essential oil of *D. japonicus* flowering aerial parts can play an important role in stored-grain protection and

Table II. Contact toxicity of the essential oil of *D. japonicus* and its main compounds against *S. zeamais* (SZ) and *T. castaneum* (TC) adults.

Insect	Treatment	Content (%)	LD ₅₀ [μ g/adult]	95% FL	Slope $\pm$ SE	Chi square (χ^2)
SZ	Essential oil	5.31–30.00	18.32	16.47–20.36	5.89 $\pm$ 0.86	23.8
	1,8-Cineole	7.43–28.57	16.34	14.87–17.76	2.71 $\pm$ 0.27	15.49
	<i>trans</i> -Geraniol	1.81–28.57	15.11	13.85–16.45	4.87 $\pm$ 0.4	14.56
	Linalool	5.31–40.00	24.88	22.90–27.38	2.34 $\pm$ 0.21	8.12
	Pyrethrum extract*	–	4.29	3.86–4.72	–	–
TC	Essential oil	5.92–30.00	13.45	15.31–18.45	4.99 $\pm$ 0.61	17.36
	1,8-Cineole	3.95–20.00	12.67	11.56–13.79	3.56 $\pm$ 0.31	12.11
	<i>trans</i> -Geraniol	5.92–30.00	21.34	19.44–23.47	2.89 $\pm$ 0.24	0.987
	Linalool	2.63–20.00	8.12	7.43–9.09	3.12 $\pm$ 0.36	12.32
	Pyrethrum extract*	–	0.36	0.32–0.41	–	–

* Data from Liu *et al.* (2010).Table III. Fumigant toxicity of the essential oil of *D. japonicus* and its main compounds against *S. zeamais* (SZ) and *T. castaneum* (TC) adults.

Insect	Treatment	Content (%)	LC ₅₀ [mg/l air]	95% FL	Slope $\pm$ SE	Chi square (χ^2)
SZ	Essential oil	2.20–18.00	10.11	21.99–28.69	2.87 $\pm$ 0.31	15.36
	1,8-Cineole	0.95–10.00	2.96	2.76–3.21	1.72 $\pm$ 0.15	9.48
	<i>trans</i> -Geraniol	1.86–15.00	15.11	13.85–16.45	4.87 $\pm$ 0.47	14.56
	Linalool	1.32–10.00	10.02	9.01–11.29	2.67 $\pm$ 0.28	12.45
	MeBr*	–	0.67	–	–	–
TC	Essential oil	1.80–20.00	5.26	4.74–5.86	2.48 $\pm$ 0.26	8.12
	1,8-Cineole	1.32–15.00	4.68	4.17–5.06	3.14 $\pm$ 0.33	15.85
	<i>trans</i> -Geraniol	2.60–15.00	14.68	13.37–15.64	3.12 $\pm$ 0.41	15.85
	Linalool	1.32–10.00	9.34	8.64–10.17	2.89 $\pm$ 0.32	8.64
	MeBr*	–	1.75	–	–	–

* From Liu and Ho (1999).

reduce the need for synthetic insecticides and the associated risks. However, for the practical application of the essential oil as novel insecticide/fumigant, further studies on the safety of the essential oil to humans and on the development of formulations are necessary to improve the efficacy and stability and to reduce costs.

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- Adams R. P. (2007), Identification of Essential Oil Components by Gas Chromatography/Mass Spectrometry, 4th ed. Allured Publishing Corporation, Carol Stream, IL, USA.
- Arabi F., Moharrampour S., and Sefidkon F. (2008), Chemical composition and insecticidal activity of essential oil from *Perovskia abrotanoides* (Lamiaceae) against *Sitophilus oryzae* (Coleoptera: Curculionidae) and *Tribolium castaneum* (Coleoptera: Tenebrionidae). *Int. J. Trop. Insect Sci.* **28**, 144–150.
- Chinese Pharmacopoeia Commission (2010), The Pharmacopoeia of the People's Republic of China. Chemical Industry Press, Beijing, China, Vol. 1, pp. 131–132.
- Chu S.-S., Liu Q.-R., and Liu Z.-L. (2010a), Insecticidal activity and chemical composition of the essential oil of *Artemisia vestita* from China against *Sitophilus zeamais*. *Biochem. Syst. Ecol.* **38**, 489–492.
- Chu S.-S., Liu S.-L., Jiang G.-H., and Liu Z.-L. (2010b), Composition and toxicity of essential oil of *Illicium simonsii* Maxim. (Illiciaceae) fruit against the maize weevils. *Rec. Nat. Prod.* **4**, 205–210.
- Flora of China Editorial Board (1986), The Flora of China. Sciences Press, Beijing, China, Vol. 73(1), pp. 62–63.
- Isman M.-B. (2006), Botanical insecticides, deterrents, and repellents in modern agriculture and an increasingly regulated world. *Annu. Rev. Entomol.* **51**, 45–66.
- Jiangsu New Medical College (1977), Dictionary of Chinese Herbal Medicine. Shanghai Science & Technology Press, Shanghai, China, pp. 2267–2269.
- Lee B.-H., Annis P.-C., Tumaalii T., and Choi W.-C. (2004), Fumigant toxicity of essential oils from the Myrtaceae family and 1,8-cineole against 3 major stored-grain insects. *J. Stored Prod. Res.* **40**, 553–564.
- Li H.-Q., Bai C.-Q., Chu S.-S., Zhou L., Du S.-S., Liu Z.-L., and Liu Q.-Z. (2011), Chemical composition and toxicities of the essential oil derived from *Kadsura heteroclita* stems against *Sitophilus zeamais* and *Meloidogyne incognita*. *J. Med. Plants Res.* **5**, 4943–4948.
- Liu Z.-L. and Ho S.-H. (1999), Bioactivity of the essential oil extracted from *Evodia rutaecarpa* Hook. f. et Thomas against the grain storage insects, *Sitophilus zeamais* Motsch. and *Tribolium castaneum* (Herbst). *J. Stored Prod. Res.* **35**, 317–328.
- Liu Z.-L. and Du S.-S. (2011), Fumigant components from the essential oil of *Evodia rutaecarpa* Hort unripe fruits. *E-J. Chem.* **8**, 1937–1943.
- Liu Z.-L., Liu Q.-R., Chu S.-S., and Jiang G.-H. (2010), Insecticidal activity and chemical composition of the essential oils of *Artemisia lavandulaefolia* and *Artemisia sieversiana* from China. *Chem. Biodivers.* **7**, 2040–2045.
- Magan N., Hope R., Cairns V., and Aldred D. (2003), Postharvest fungal ecology: impact of fungal growth and mycotoxin accumulation in stored grain. *Eur. J. Plant Pathol.* **109**, 723–730.
- Miao Z.-C., Feng R., Zhou Y.-X., and Wei F. (2000a), Chemical structure and NMR of a new saponin from *Dipsacus japonicus* (Dipsacaceae). *Acta Bot. Sin.* **42**, 421–426.
- Miao Z.-C., Zhou Y.-X., Feng R., and Wei F. (2000b), Structural determination of a new bidesmosidic triterpenoid glycoside from *Dipsacus japonicus*. *Chin. J. Org.* **20**, 81–87.
- Mohamed M.-I. and Abdelgaleil S.-A.-M. (2008), Chemical composition and insecticidal potential of essential oils from Egyptian plants against *Sitophilus oryzae* (L.) (Coleoptera: Curculionidae) and *Tribolium castaneum* (Herbst) (Coleoptera: Tenebrionidae). *Appl. Entomol. Zool.* **43**, 599–607.
- Rajendran S. and Srianjini V. (2008), Plant products as fumigants for stored-product insects control. *J. Stored Prod. Res.* **44**, 126–135.
- Sakuma M. (1998), Probit analysis of preference data. *Appl. Entomol. Zool.* **33**, 339–347.
- Suthisut D., Fields P.-G., and Chandrapatya A. (2011), Fumigation toxicity of essential oils from three Thai plants (Zingiberaceae) and their major compounds against *Sitophilus zeamais* and *Tribolium castaneum*. *J. Stored Prod. Res.* **47**, 222–230.
- Trinh T.-T., Tran V.-S., and Adam G. (1999), Study on chemical constituents of *Dipsacus japonicus*. I. Iridoid and bis-iridoid glycosides. *Vietnam J. Chem.* **37**, 64–69.
- Trinh T.-T., Tran V.-S., and Adam G. (2002), Study on chemical constituents of *Dipsacus japonicus*. II. Triterpene glycosides. *Vietnam J. Chem.* **40**, 13–19.
- Tripathi A.-K., Prajapati V., Aggarwal K.-K., and Kumar S. (2001), Toxicity, feeding deterrence, and effect of activity of 1,8-cineole from *Artemisia annua* on progeny production of *Tribolium castaneum* (Coleoptera: Tenebrionidae). *J. Econ. Entomol.* **94**, 979–983.
- Wei F., Lou Z. C., Gao M., and Miao Z. C. (1995), Application of new techniques of NMR in structure elucidation of japonidipsaponin E₁ isolated from *Dipsacus japonicus* Miq. *Acta Pharm. Sin.* **30**, 831–837.
- Wei F., Liu L.-M., and Lou Z.-C. (1998), Application of new techniques of NMR in structure elucidation of japonidipsaponin E₂ isolated from *Dipsacus japonicus* Miq. *J. Shenyang Pharm. Univ.* **15**, 120–124.
- Zapata N. and Smagghe G. (2010), Repellency and toxicity of essential oils from the leaves and bark of *Laurelia sempervirens* and *Drimys winteri* against *Tribolium castaneum*. *Ind. Crops Prod.* **32**, 405–410.
- Zettler J.-L. and Arthur F.-H. (2000), Chemical control of stored product insects with fumigants and residual treatments. *Crop Prot.* **19**, 577–582.