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BIO-INSECTICIDAL EFFICACY OF FOUR ESSENTIAL OILS AGAINST ADULTS OF SITOPHILUS GRANARIUS L. (COLEOPTERA: CURCULIONIDAE) AND LARVAE OF TENEBRIO MOLITOR (COLEOPTERA: TENEBRIONIDAE)

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Bio-insecticidal efficacy of four essential oils against adults of Sitophilus granarius (Coleoptera: Curculionidae) and larvae of Tenebrio molitor (Coleoptera: Tenebrionidae). – Baranová B., Gruľová D. – *The bio-insecticidal efficacy of essential oils from anise (Pimpinella anisum L.), cumin (Carum carvi L.), fennel (Foeniculum vulgare Miller) and origanum (Origanum vulgare L.) essential oils was tested using adults of wheat granary weevil, Sitophilus granarius L. (Coleoptera: Curculionidae) and larvae of yellow mealworm, Tenebrio molitor L. (Coleoptera: Tenebrionidae), as the two representants of stored grain pests. From the essential oils used, cumin showed the highest bio-insecticidal efficacy by both types of tests. From the model organisms used, only adults of wheat granary weevil were affected, since yellow mealworm larvae did not respond nor to any of the essential oils used neither to test type. Essential oils seem to have strong potential to replace dangerous synthetic toxic insecticides, however, their efficacy seems to be strongly dependent on the target organisms as well as way of application.*
Keywords: *wheat granary weevil, Sitophilus granarius, yellow mealworm, Tenebrio molitor, bio-insecticides, plant extract, essential oil, anise, cumin, fennel, origanum.*
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Біоінсектицидна ефективність чотирьох ефірних олій щодо імаго Sitophilus granarius (Coleoptera: Curculionidae) та личинок Tenebrio molitor (Coleoptera: Tenebrionidae). – Баранова Б., Ґрульова Д. – *Ефірні олії, як вторинні метаболіти рослин, містять широкий спектр біологічно активних сполук, які можуть впливати на метаболічні, біохімічні, фізіологічні та поведінкові функції комах. Метою даного дослідження було оцінити фумігантну і контактну токсичність ефірних масел деяких видів рослин, що використовуються в кулінарії, порівняти ефективність різної дози та часу експозиції, оцінити різницю між відпрацьованими оліями у відсотках смертності, визначити основні сполуки використовуваних ефірних масел. Біоінсектицидну активність ефірних олій анісу (Pimpinella anisum L.), кмину (Carum carvi L.), фенхеля (Foeniculum vulgare Miller) та материнки (Origanum vulgare L.) досліджували на модельних об'єктах. В якості останніх були обрані імаго довгоносика комірного – Sitophilus granarius L. (Coleoptera: Curculionidae) та личинки хрущака борошняного – Tenebrio molitor L. (Coleoptera: Tenebrionidae). З випробуваних ефірних олій, олія з кмину проявила найвищу біоінсектицидну активність. Модельні організмів по різному реагували на вплив еферних олій. Були уражені лише імаго Sitophilus granarius, натомість личинки Tenebrio molitor не реагували на жодний вид олій, які використовували в тестових випробуваннях. З метою заміщення небезпечних токсичних фумігантів та інсектицидів ефірні олії є можливим інструментом захисту сільськогосподарських культур та продуктів харчування від комах-шкідників, як безпечна альтернатива для людини та довкілля, можуть бути використані в сільському господарстві в системі метоідв інтегрованого захисту від комах-шкідників. Однак, ефективність ефірних олій суттєво залежить від цільових груп організмів, а також способу застосування.*
Ключові слова: *довгоносик комірний, Sitophilus granarius, хрущак борошняний, Tenebrio molitor, біоінсектициди, рослинні екстракти, ефірна олія, аніс, кмин, фенхель, материнка.*
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Introduction

Essential oils, as the secondary plant metabolites, dispose with a wide spectrum of biologically active compounds. Production of aromatic oils is a part of the defence system, protecting plants against herbivores, plant juice sucking insect, viruses, microbes or fungi. Equally, essential oils can affect metabolic, biochemical, physiological or behavioural functions of the insect representatives. These features make essential oils to be possible tools for crops and stored products protection against insect pest, as the plant-based, health and ecologically safely alternative approaches in agriculture (Raguso, Pichersky 1999; Das et al. 2013; Campolo et al. 2018).

Adults of wheat granary weevil, *Sitophilus granarius* Linnaeus, 1758 (Coleoptera: Curculionidae) and larvae of yellow mealworm, *Tenebrio molitor* Linnaeus, 1758 (Coleoptera: Tenebrionidae), which belongs to the most common as well as the most harmful grain pests.

Adults of wheat granary weevil cause damage of wheat, barley, oat, rice or maize grains and products, as feeding upon them, or laying down the eggs. Larvae of yellow mealworm damage different fractured grains of maize, wheat or soybean, their metabolism products cause grains contamination (Martynov et al. 2019).

Different spectrum of essential oils was tested for their bio-insecticidal efficacy against *Sitophilus* genus representatives as well as *Tenebrio molitor* larvae, including also anise, cumin, fennel and origanum oils used in our study.

Karakas and Bolukbasi (2017) tested cumin EO biological activity against *Sitophilus granarius*. Ziaee et al. (2015) pointed importance of the temperature for the efficiency of the cumin essential oil against granary weevil in the integrated pest management programs of stored products. Fang et al. (2010) observed strong contact toxicity of EO from *Carum carvi* fruits against *Sitophilus zeamais* (Motsch., 1855). Fenel EO was tested for its larvicidal activity against *Tenebrio molitor* by Cossimi et al. (2009). Origanum adulticidal efficacy was tested against *Sitophilus oryzae* (L., 1763) (Karan, Simsek 2018; Abdelgaleil et al. 2019) and *Sitophilus zeamais* (Bittner et al. 2008). Study of Alkan (2020) determined contact and fumigant activity of *Origanum vulgare* against granary weevil adults (Martynov et al. 2019).

Expect entire essential oils, also their major compounds were tested for their bio-insecticidal activity: fumigant activity of anethole as the major compound of *Pimpinella anisum* and fennel essential oils was tested using *Sitophilus oryzae* (Tunc, Erler, 2000). Adulticidal efficacy of cumin main compound carvone was tested against *Sitophilus zeamais* and *Sitophilus granarius* under laboratory conditions (Vejražka et al. 2007; Yildirim et al. 2013).

The aim of our study was to:

a) evaluate the fumigant and contact toxicity of anise, cumin, fennel and origanum essential oils against adults of granary wheat weevil and yellow mealworm larvae;

b) compare efficacy of the different dose and time of exposition;

c) assess distinction between used oils in the mortality percentage;

d) determine main compounds of the essential oils used.

Material and methods

Essential oils

Essential oils from anise (*Pimpinella anisum* L.), cumin (*Carum carvi* L.), fennel (*Foeniculum vulgare* Miller) as representatives of *Apiaceae* family and origanum (*Origanum vulgare* L.) as the representative of *Lamiaceae* family were tested. EOs were obtained from a private company of Calendula, a.s., Nová Ľubovňa, Slovakia. The oils were stored in brown glass bottles at 5°C until the use.

Analysis of the EO´s composition was done as follows: pure EOs were diluted to 1:1000 ratio in n-hexane and subjected to analysis by gas chromatography - mass spectrometry (GC-MS) as described in Gruľová et al. (2020), gas chromatography - mass spectrometry (GC-MS). Identification of the main compounds of EOs was provided using library NIST 02 and by comparison of the Rt with external standards.

Pure, non-diluted EOs were used in the experiment.

Model organisms

Two different beetle representants of stored grain pests were used in the study: adults of wheat granary weevil, *Sitophilus granarius* Linnaeus, 1758 (Coleoptera: Curculionidae) and larvae of yellow mealworm, *Tenebrio molitor* Linnaeus 1758 (Coleoptera: Tenebrionidae).

Wheat granary weevil adults were bred at the University of Prešov, Slovakia. Specimens were reared in a 1 L wide-mounted glass jars, half filled up with wheat grains. Mouth of the jars was covered with a fine mesh cloth insuring ventilation and to prevent beetles escaping. Jars were kept at average temperature 22 ºC and by 55% relative humidity. Mealworms were obtained from commercial breeder and kept in darkness at room temperature (22°C) and by 55% relative humidity in plastic box with feeding substratum (wheat flour / bread-crumb / semolina – 1:1:1) till experiment. All following experiments were carried out under the same conditions (22 ºC, 55% relative humidity).

Fumigant test - time and dose response bioassay

To test fumigant toxicity, aliquots of 250 µl and 750 µl of each from essential oils (*Pimpinella anisum*, *Carum carvi*, *Foeniculum vulgare*, *Origanum vulgare*) were pippeted into the 1.5 ml Eppendorf tube with penetrated lids. Tubes were fixed with transparent duct tape into the centre of glass Petri dishes with 90 mm diameter x 15 mm height, with overall volume of 127 ml, what means effective doses of 1.97 µl/ml and 5.91 µl/ml of air respectively. Ten adults of the wheat granary weevil were placed together with 1 gram of wheat grain in the dishes. Petri dishes were then closed, sealed with Parafilm to prevent beetles escaping and transferred into a chamber room at 20±2°C, 55% relative humidity and dark regime. Identical treatment was done with ten larvae of yellow mealworm, but without grains. All of the treatments were replicated three times. The control treatments did not include essential oil nor grains. The number of dead *S. granarius* adults / *T. molitor* larvae was determined at 24, 48 and 72 h after the initiation of the treatment. The weevil adults were considered dead if their appendages did not move when proved with a fine brush, mealworm larvae if shoving no movement after being softly squeezed behind head with soft pincette.

Contact toxicity test- dose response bioassay

To test contact efficacy, amounts 2.5 µl, 5 µl, 9.5 µl and 14 µl of each from essential oils (*Pimpinella anisum*, *Carum carvi*, *Foeniculum vulgare*, *Origanum vulgare*) were pippeted on 1×1 cm piece of filter paper placed in the cells of plastic, rectangular cultures plates. Plates of 12.5×8.5×1.8 cm (w/l/h), with 6 cells of 3.1 cm diameter were used for *Tenebrio molitor* larvae, for *Sitophilus granarius* adults, plates of 12.5×8.5×1.8 cm (w/l/h) with 12 cells were used, when the cell was of 2.3 cm diameter.

Three larvae of *Tenebrio molitor*/ten adults of *Sitophilus granarius* were placed in the one cell. Whole plates were then sealed with Parafilm, closed and transferred into a chamber room at 20±2°C, 55% relative humidity and dark regime. All of the treatments were replicated three times. The control treatments did not include essential oil. The number of dead *S. granarius* adults/*T. molitor* larvae was determined 48h after the initiation of the treatment similarly, as mentioned above. As the essential oils have a tendency to evaporate, we strongly take into account, that the test is a like combination of contact as well as fumigant toxicity.

Data analysis

The bio-insecticidal efficacy of essential oils was expressed through mortality %, i.e. % of the dead model organisms within the experiment from the whole amount of model organisms used. Mean values were determined using Univariate statistics, Descriptive Statistic was used to depict observed distinctions (PAST 2.17c, Hammer et al. 2001). Student T-TEST in excel was used to evaluate distinctions in the % of dead model organisms between the experiment and control, between particular concentrations of the same essential oil as well as between the different essential oils used, with three levels of significance ($p \leq 0.05$; 0.01; 0.001). Lethal doses LD50 and lethal times LT50 were estimated based on the Probit analyse.

Results

Fumigant toxicity

Fumigant toxicity of the used essential oils against *Sitophilus granarius* adults decreased in the following order: cumin > fennel > anise > origanum. Essential oil of *Carum carvi* showed in general the strongest fumigant toxicity from the four EOs used in the experiment, and significantly ($p \leq 0.05$) higher fumigant toxicity in comparison to anise and origanum EOs by 250 µl dose. By 750 µl dose, fumigant toxicity of used EOs was balanced as we did not observe any differences in the mortality % between the oils.

Expect origanum (250 µl dose), EOs caused significantly higher mortality of granary weevil adults in comparison to control (Table 1).

Table 1. Fumigant toxicity of the different effective doses (ED = µl of EO per ml of air) of essential oils against adults of wheat granary weevil

Mortality (%) *Sitophilus granarius* adults, fumigant test						
ED/ToE	1.97/24	1.97/48	1.97/72	5.91/24	5.91/48	5.91/72
anise	40.58±17.30**	61.08±27.82**	68.67±24.42**	64.5±34.23***	87±22.14***	94.67±18.24**
cumin	79±25.03***	95±122.25***	98±4.08***	100***	100***	100***
fennel	47±14.81***	72±13.22***	87.83+11.97**	75.44±25.05**	88.07±25.04**	98.48±3.71***
origanum	12.17±18.98	19.17±24.37	37.96±44.50	41.08±27.14**	61±41.04**	90±24.50***
control	0/24 h		1.67±4.08/48 h		5±8.37/72 h	

Note: different time of exposition (ToE in hours), expressed as mortality % ± standard deviation (SD). Significant distinction in % of dead model organisms between experiment and control evaluated using Student T-TEST is indicated as follows: ** $p<0.01$; *** $p<0.001$.

We did not observe any differences between effective doses (250 µl/750 µl) in the fumigant toxicity based on the mortality (%) of *Sitophilus granarius* adults by anise and cumin essential oils. By fennel EO, 750 µl dose showed significantly ($p \leq 0.05$) higher fumigant toxicity in comparison to 250 µl dose after 24 h exposition, however, after 48 h and 72 h no differences were observed.

By origanum EO, no differences were observed after 24 h and 48 h exposition, but, after 72 h, 750 µl dose was confirmed as significantly ($p<0.05$) more effective in comparison to 250 µl.

Generally, 750 µl showed significantly ($p<0.05$) higher fumigant toxicity in comparison to 250 µl dose by every from essential oils used, and the mortality increased with the time of exposition (Fig. 1). Exposition times causing 50% mortality of wheat granary weevil adults are listed in Table 2.

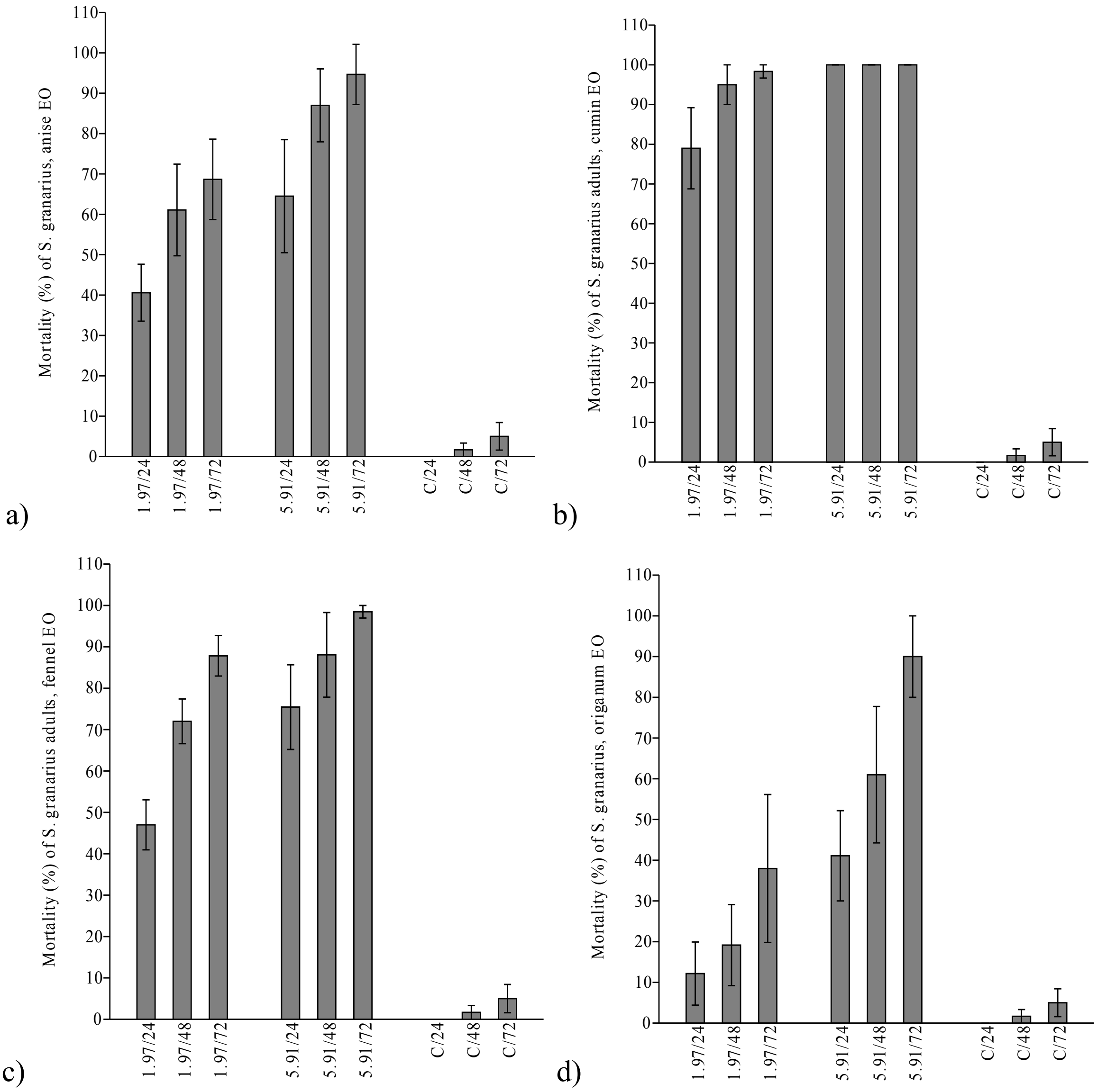

Fig. 1. Fumigant toxicity of the different amounts of anise (a), cumin (b), fennel (c) and origanum (d) essential oils against adults of the granary weevil evaluated after 24 h, 48 h and 72 h period in comparison to control.

Table 2. LT50 according to Probit analyse for two effective doses of essential oils used

LT50 / hours	anise	cumin	fennel	origanum
1.97 µl of EO/ml of air	33 h	12 h	27 h	128 h
5.91 µl of EO/ml of air	17.5 h	-	19.15 h	30.45 h

We did not observe any fumigant toxicity of used EOs against larvae of *Tenebrio molitor*.

Contact toxicity activity

Contact toxicity of used essential oils against *Sitophilus granarius* adults generally decreased in the following order: cumin > origanum > fennel > anise. Significant distinctions in mortality % between experiment and control were observed only by cumin and origanum EOs (Table 3).

Table 3. Contact toxicity of the different amounts of essential oils against adults of wheat granary weevil after 48 hours exposition expressed as mortality % ± standard deviation (SD).

	Mortality (%) of *Sitophilus granarius* adults, 48 h			
EO amount	2.5 µl	5 µl	9.5 µl	14 µl
anise	30±20	12.5±12.5	22±9.85	25±5
cumin	100***	100***	100***	100***
fennel	16.67±11.55	20.67±10.07	24.33±7.51	33.67±24.71
origanum	96.67±5.77***	96.67±5.77***	100***	100***
control	10±10			

Note: significant distinction between experiment and control in % of dead model organisms evaluated using Student T-TEST is indicated as follows: ***p<0.001.

Essential oils of *Carum carvi* and *Origanum vulgare* showed in general the strongest contact toxicity against wheat granary weevil adults from the four EOs used and significantly ($p \leq 0.05$) higher contact toxicity in comparison to anise and fennel EOs by every oil amount.

Cumin and origanum essential oils contact toxicity did not change according to the oil amount applied. By fennel EO, with increasing amount of essential oil used, there increased also the mortality of model organisms, however, there were no distinctions between the % of the dead granary weevil adults as comparing particular EO´s amounts. By anise EO, the highest mortality was observed by the lowest dose, consequently, similarly to fennel EO, the number of dead *S. granarius* increased with increasing amount of essential oil applied, but no distinctions were observed between particular effective doses (Fig. 2).

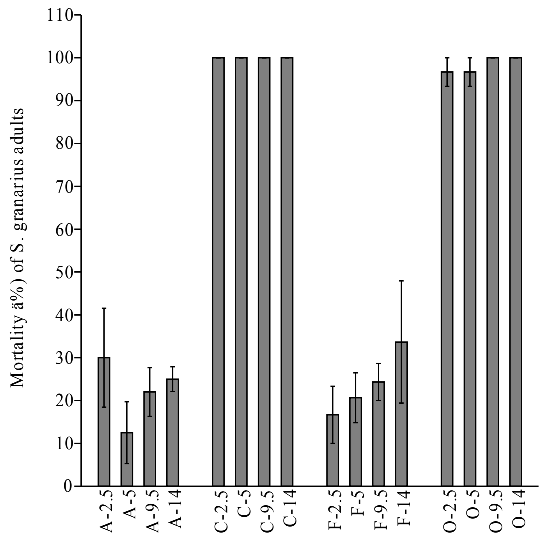

Fig. 2. Contact toxicity of the different amounts (2.5 µl, 5 µl, 9.5 µl, 14 µl) of essential oils against adults of granary weevil evaluated after 48h period. Abbreviations and notes: A-anise, C-cumin, F-fennel, O-origanum.

According to Probit analyse, LD50 doses were as follows: anise 13 µl , fennel 23.5 µl and origanum 0.20 µl for 48 hours of exposition.

We did not observe any contact toxicity of used EOs against larvae of *Tenebrio molitor*.

Representation of the main compounds of the four essential oils used in the experiment are listed in the Table 4, since according their chemical composition, all listed compounds represent monoterpenoids.

Table 4. Dominant components (>15%) of essential oils used in the experiment determined using GC-MS analyse

component	anise	cumin	fennel	origanum
Limonene		26.4		
Fenchone			16.5	
Estragole			40.1	
Carvone		70.2		
Anethole	88.6		38.1	
Carvacrol				78.2

Discussion

From the essential oils used in our study, EO from *Carum carvi* showed the strongest bio-insecticidal efficacy in the both tests against wheat granary weevils, what is in accordance with Vejražka et al. (2007), Fang et al. (2010) or Karakas and Bolukbasi (2017). We identified also the same main compounds, i.e. carvone and limonene in cumin EO, what support idea, that the monoterpenoids have significantly insecticidal effect (Vejražka et al., 2007; Yildirim et al., 2010), however, according to our results, their bio-insecticidal efficacy is variable.

Concerning origanum EO, we observed ambivalent results, as the fumigant toxicity was the lowest from the essential oils used, but, contact toxicity was strong, what is in accordance with Alkan (2020) results. *Origanum vulgare* EO bio-insecticidal efficacy can be connected with carvacrol as its main compound (Erler, 2005; Kim and Park, 2008).

Anise and fennel essential oils showed bio-insecticidal efficacy only by fumigant test. By contact toxicity test, to achieve 50% mortality of model organisms, the dose should be fourfould as the highest amount (14 µl) used by anise EO, and even seven-times higher by fennel EO, according to Probit analyse.

In general, bio-insecticidal efficacy increased as the dose of essential oil increased, as well as the time of exposition elongated, or, that the effective time of exposition shortens, if the higher amount of EO is used.

Two different beetles representants of stored grain pests were used in the study. Because practically zero bio-insecticidal efficacy of used essential oils was observed against yellow mealworm larvae, EO´s biological activity obviously strongly depend on the model, i.e. target organisms, development phase (juvenile/imago) and the way of application of essential oils, however, also *Tenebrio molitor* is obviously susceptible to EO´s insecticidal activity (Plata-Rueda et al., 2017). Thus, granary weevil adults seem to by significantly susceptible to the effect of essential oils in comparing to yellow mealworm larvae.

Conclusion

As a result of the study, used essential oils showed both, contact and fumigant toxicity, since the bio-insecticidal efficacy varies according to time of exposition, model, i.e. target organism used, effective dose and essential oil composition. Nevertheless, in aim to replace dangerous and toxic fumigants and insecticides essential oils seem to be possible tools for crops and stored products protection against insect pest, as the human and ecological safely alternative in agriculture and integrated pest management.

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