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ORIGINAL RESEARCH ARTICLE

Antifeedant, growth regulatory and biochemical effects of terpenes and phenylpropenes on *Spodoptera littoralis* Boisduval

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Abstract

Screening the biological activities of plant secondary metabolites on economic pests can lead to discovery new ecofriendly biopesticides. The aim of this work was to evaluate the antifeedant, growth inhibitory and toxic activities of seven monoterpenes, two phenylpropenes and two sesquiterpenes on 2nd larval instar of *Spodoptera littoralis*. The tested compounds induced a significant antifeedant effect at various concentrations (500, 1000 and 2000 mg/kg), particularly after 6 and 9 days of exposure. Among the tested compounds, *trans*-cinnamaldehyde, α-terpinene, (−)-citronellal and 1,8-cineole were the most potent antifeedants after the three exposure periods. In general the tested compounds showed remarkable antifeedant activity after 9 days of exposure as their antifeedant indices ranged between 44.0 and 80.1%. On the other hand, the tested compounds drastically inhibited the growth of *S. littoralis* larvae at the tested concentrations. The larval growth inhibition ranged between 21.4 and 100% with cuminaldehyde, 1,8-cineole and eugenol being the most potent growth inhibitors. Some of the tested compounds caused significantly higher antifeedant and growth inhibitory effects than a reference insecticide, pyriproxifen. In general, the tested compounds showed higher growth inhibition than antifeedant effect. The tested compound also induced *S. littoralis* larval morality which improved with increasing exposure time and concentration. Cuminaldehyde, 1,8-cineole and (−)-carvone showed highest toxicity with 100.0, 97.0 and 77.0% mortality, respectively, at 2000 mg/kg after 9 days of exposure. Biochemical studies revealed that *trans*-cinnamaldehyde (IC_{50} = 0.03 mM), farnesol (IC_{50} = 0.04 mM) and eugenol (IC_{50} = 0.06 mM) are potent α-amylase inhibitors. These three compounds also caused significant inhibition of total proteases activity. This is the first report on antifeedant, growth inhibitory and insecticidal activities of the tested compounds on *S. littoralis*. Moreover, the strong bioactivity reported in this study indicated that these compounds have a potential to be used as bioinsecticides.

Keywords Monoterpenes · Phenylpropenes · Sesquiterpenes · Biological activity · α-Amylase · Total proteases · *Spodoptera littoralis*

Introduction

Developing new biopesticides from plant-derived products is an important issue in modern agricultural production systems. Recently, many countries around world have headed towards the use of integrated pest management (IPM) programs due to the several drawbacks associated with continuous use of synthetic pesticides (Czaja et al. 2015; Tonial et al. 2017). One of the main components of IPM is the application of plant extracts, oils and secondary metabolites in pest control as class of biopesticides. In this regard, plant products have been used as efficient toxicants, antifeedants, repellents and growth regulators against economic agricultural and public health insects (Miresmailli and Isman 2014; Pavela 2016; Szczepanik et al. 2016; Hernández-Carlos and Gamboa-Angulo 2019).

Certain classes of plant secondary metabolites, particularly monoterpenes, phenylpropenes and sesquiterpenes, are viewed as exceptionally promising natural pesticides. These three classes of compounds are usually present as major constituents in plant essential oils. These compounds have several ecological functions in plants, such as protection against insects, animals and pathogens, attraction of pollinators and

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allelopathy (Fischer et al. 1994; Langenheim 1994; Dudareva and Pichersky 2008). In addition, several studies have reported the wide spectrum of biological activities of monoterpenes, phenylpropenes and sesquiterpenes on economic insects as they can act as insecticides (Abdelgaleil et al. 2009; Wu et al. 2016; Saad et al. 2018), antifeedants (Gonzalez et al. 1997; Rajkumar et al. 2019), repellents (Watanabe et al. 2005; Peixoto et al. 2015), insect growth regulators (Céspedes et al. 2001; Zahran and Abdelgaleil 2011).

The cotton leafworm, *Spodoptera littoralis* (Boisduval), is among the most damaging lepidopterous insects to several important crops in subtropical and tropical zones. It attacks about 87 plant species belong to more than 40 families (Capinera 2008). Because of its highly polyphagous behavior, it is considered as a devastating insect. Therefore, some strategies have been developed to reduce the economic damage caused by *S. littoralis*, including the use of biorational chemical control and IPM.

Few reported studies were found in the literature on the antifeedant and growth inhibitory effects of monoterpenes, phenylpropenes and sesquiterpenes against *S. littoralis* (Gonzalez et al. 1997; Zapata et al. 2009; Ali et al. 2017). Therefore, the present study aimed to evaluate the antifeedant, growth inhibitory and toxic effects of seven monoterpenes [cuminaldehyde (major component of cumin oil), (−)-carvone (major component of caraway oil), (−)-citronellal (major component of citronella oil), 1,8-cineole (major component of eucalyptus oil), (−)α-pinene (major component of pine tree oil), α-terpinene (major component of citrus oils) and *p*-cymene (major component of cumin and thyme oils)], two phenylpropenes [*trans*-cinnamaldehyde (major component of cinnamon oil) and eugenol (major component of clove oil)] and two sesquiterpenes [(farnesol (component of lemon grass, citronella and other oils) and (*Z,E*)-nerolidol (major component of neroli and nerolina oils)] against the second larval instar of *S. littoralis*. Also, the inhibitory effects of selected compounds on the activity of two digestive enzymes, α-amylase and total proteases, were studied.

Materials and methods

Test insect

A susceptible strain of cotton leafworm, *Spodoptera littoralis* Boisduval (Lepidoptera: Noctuidae) was kept under laboratory conditions at 26 ± 2 °C and 70 ± 5% RH. The larvae were fed on *Ricinus communis* L. (Euphorbiaceae) leaves as described by El-Defrawi et al. (1964). The second-larval instar of *S. littoralis* was chosen in this study because it is the first damaging larval stage. Also, this stage takes more time to reach the pupal stage than third and fourth stages that allow measuring antifeedant, growth inhibitory and toxic effects after 6 and 9 days of treatment.

Test compounds

Seven monoterpenes, two phenylpropenes and two sesquiterpenes were purchased from Sigma–Aldrich Chemical Co., Steinheim, Germany, and used in this study. The compounds are cuminaldehyde (98%), (−)-carvone (98%), (−)-citronellal (95%), 1,8-cineole (99%), (−)α-pinene (98%), α-terpinene (85%), *p*-cymene (99%), *trans*-cinnamaldehyde (99%), eugenol (99%) farnesol (95%) and (*Z,E*)-nerolidol (98%). Chemical structures of these compounds are shown in Fig. 1. A reference insecticide, pyriproxyfen (98%), was obtained from Kafr El-Zayat Pesticides and Chemicals Co., Egypt. All solvents and reagents used in experiments were of high performance liquid chromatography (HPLC) grade.

Antifeedant, growth inhibition and toxicity assay

Monoterpenes, phenylpropenes and sesquiterpenes were evaluated for their antifeedant, growth inhibitory and toxic effects on the second-larval instar of *S. littoralis* by using diet-non-choice method (Abdelgaleil and El-Aswad 2005). The solutions of compounds were first prepared in acetone and incorporated with the artificial diet (Bakry et al. 1973) to give concentrations of 500, 1000 and 2000 mg/kg. Control treatment was diet mixed acetone at concentration of 0.5% (*v*/*w*). After complete evaporation of the solvent, 10 g of treated diet was placed in each Petri dish (9.0 cm diameter). Then 10 preweighed second larval instars were introduced to each Petri dish. Five replicates were carried out for each concentration. The eaten diet by each larva was determined after 3, 6 and 9 days of feeding by weighing the remaining diet in each Petri dish. Then, antifeedant index was calculated by the equation:

$$\text{Antifeedant index} = [(\text{C}-\text{T})/\text{C}] \times 100$$

where C is the weight of diet consumed by each larva in control and T is the weight of diet consumed by each larva in the treatment (Abdelgaleil and El-Aswad 2005). The growth inhibition of larvae was assayed relative to control based on larval weight gain through 3, 6 and 9 days of feeding on the treated diet. The growth inhibition of larvae was calculated from the following equation:

$$\text{Growth inhibition} = [(\text{CL}-\text{TL})/\text{CL}] \times 100$$

where CL is the gain of larval weight in the control and TL is the gain of larval weight in the treatment (Abdelgaleil and El-Aswad 2005). The larval mortality was recorded after 3, 6 and 9 days of feeding on treated diet and the mortality percentages were calculated.

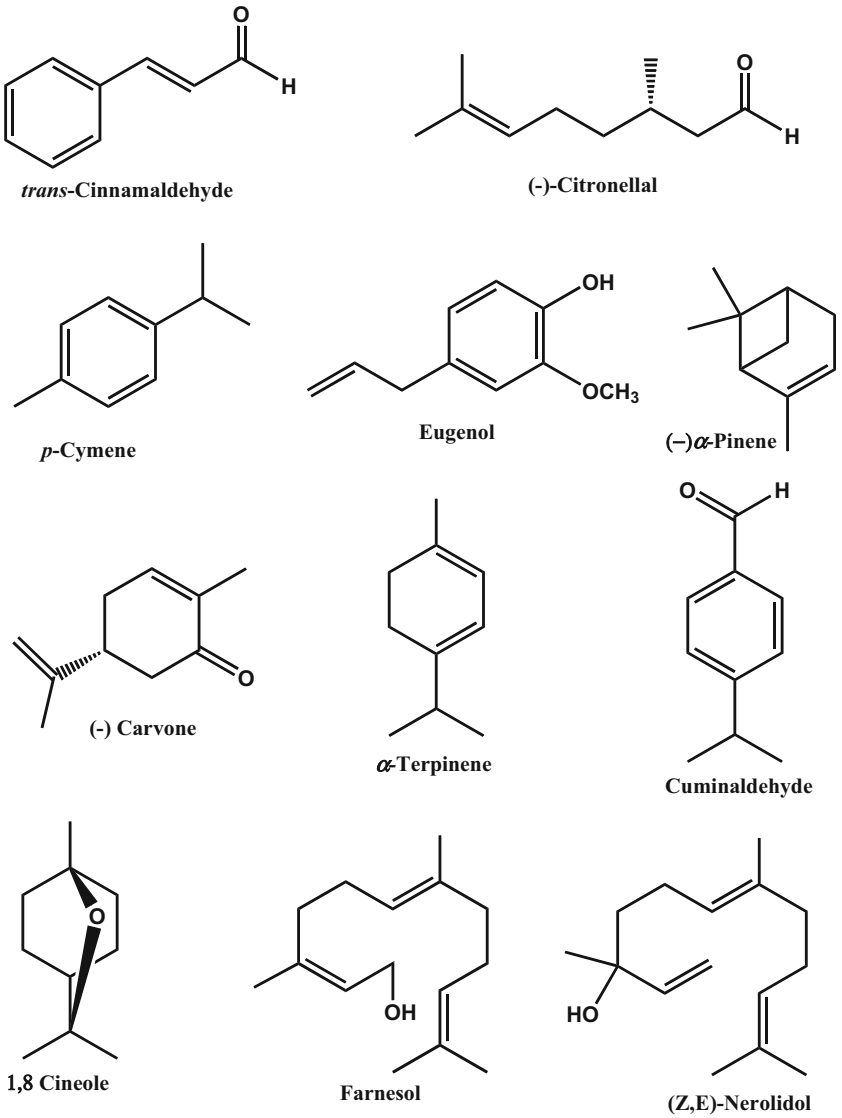

Fig. 1 The chemical structures of monoterpenes, phenylpropenes and sesquiterpenes

In vitro inhibition of α-amylase activity assay

Midguts of the 4th and 5th larval instars of *S. littoralis* were collected, excised, washed with ice-cold saline solution (0.9% NaCl) repeatedly to remove foodstuff. One gram of total larvae was homogenized in 5 ml glass distilled water using Polytron Kinemetica on ice. The homogenate was centrifuged at 15000 rpm for 15 min at 4 °C using IEC-CRU 5000 cooling centrifuge. The supernatant was used for α-amylase activity assay. The in vitro inhibition of total proteases activity was determined by incubating the enzyme for 30 min at 37 °C with different concentrations (0.005–1.0 mM) of tested compounds prepared in acetone. Emulsifying agent, Triton-X 100, was added at concentration of 0.01% to enzyme solution. The control treatments were prepared by adding 20 μl of acetone without tested compounds. Activity of α-amylase was assayed according to Kaufman and Tietz (1980). Fifty μl of enzyme source was added to an assay mixture in final volume 1 ml contains 2.3 mM 2-chloro 4-nitrophenyl-α-D-maltotrioside (CNPG3), 350 mM NaCl, 6 mM calcium acetate, 600 mM potassium thiocyanate and 100 mM Good's buffer pH 6. An assay mixture without enzyme was used as the blank. The change in absorption at 405 nm was monitored on Sequoia-Turner Model 340 spectrophotometer for 4 min. Activity of α-amylase was calculated as ΔOD_{405}/mg protein/min. The inhibition percentage of α-amylase activity was calculated. The concentrations of the tested compounds that inhibited 50% the enzyme activity (IC_{50}) were determined from a linear regression analysis (Finney 1971).

In vitro inhibition of total proteases activity assay

Midguts of the 4th and 5th larval instars of *S. littoralis* were collected, excised, washed with ice-cold saline solution (0.9% NaCl) repeatedly to remove foodstuff. Midguts were then homogenized in (1: 10 *w*/*v*) 100 mM Tris-HCl buffer pH 7 using

Polytron Kinemetica on ice. The homogenate was centrifuged at 4000 rpm for 15 min at 4 °C using IEC-CRU 5000 cooling centrifuge. The supernatant was used for total proteolytic activity estimation. The in vitro inhibition of total proteases activity was determined by incubating the enzyme for 30 min at 37 °C with different concentrations (0.02–20 mM) of tested compounds prepared in acetone. Emulsifying agent, Triton-X 100, was added at concentration of 0.01% to enzyme solution. The control treatments were prepared by adding 20 μl of acetone without tested compounds. Then, the total proteolytic activity was measured using asocasein as a substrate according to (Olga et al. 2002; Mohen and Gujar 2003). The homogenate was incubated in a total volume 60 μl of assay buffer (100 mM Tris-HCl pH 8) for 20 min at 37 °C before addition of 200 μl of 2% azocazein (*w*/*v* in assay buffer). The reaction was allowed to proceed for 180 min at 37 °C, and then stopped by addition of 300 μl cold 10% trichloroacetic acid (TCA). The reaction mixture was centrifuged at 3000 rpm for 10 min IEC-CRU 5000 cooling centrifuge. Excess acidity was neutralized by adding 10 μl NaOH (10 N) to the reaction mixture and absorbance was measured at 440 nm using Sequoia-Turner Model 340 spectrophotometer. An assay mixture without enzyme was used as the blank. The inhibition percentage of total proteases activity and IC_{50} of the tested compounds were calculated as previously described.

Statistical analysis

Significant differences among mean values of antifeedant indices, growth inhibitory and mortality percentages were determined ($P = 0.05$) by using one-way analysis of variance followed by Student–Newman–Keuls test (Cohort software Inc. 1985). The enzyme inhibition percentages were subjected to probit analysis (Finney 1971) to obtain IC_{50} values, using SPSS 21.0 (SPSS, Chicago, IL, USA).

Results

Antifeedant activity of monoterpenes, phenylpropenes and sesquiterpenes

The antifeeding activity of the monoterpenes, phenylpropenes and sesquiterpenes were tested against the 2nd larval instar of *S. littoralis* on semi-artificial diet. The tested compounds showed different levels of feed-deterrence activities at various concentrations. The antifeedant indices of the tested compounds after 3, 6 and 9 days of feeding on treated diet at concentrations of 500, 1000 and 2000 mg/kg are shown in Table 1. The antifeedant activity of tested compounds enhanced significantly with increasing the treatment period. After 3 days of feeding on treated diet, *trans*-cinnamaldehyde revealed the strongest antifeedant activity at the three tested concentrations. The antifeeant indices of this compound were 33.3, 44.4 and 44.4% at 500, 1000 and 2000, respectively. The other compounds had significantly lower antifeeant indices ranged between 1.9 and 37.0%. After 6 days, α-terpinene caused the highest feed deterrence, followed by *trans*-cinnamaldehyde, eugenol, and (−)-carvone. Their antifeedant indices were 50.0, 44.4, 38.7, and 37.9 at concentration of 500 mg/kg, respectively. In addition, 1,8-cineole revealed the highest antifeedant activity at 1000 and 2000 mg/kg with antifeedant indices of 56.5 and 78.2%, respectively. The antifeedant indices of the tested compounds ranged between 44.0 and 80.1% after 9 days of treatment. *Trans*-Cinnamaldehyde caused the highest antifeedant activity at 500 and 2000 mg/kg, while citronellal was the most potent at 1000 mg/kg. (−)-Carvone, *trans*-cinnamaldehyde and *p*-cymene were more potent antifeedant than a reference insecticide, pyriproxifen, at the tested concentrations after 3 days of treatment.

Growth inhibitory effect of monoterpenes, phenylpropenes and sesquiterpenes

The tested monoterpenes, phenylpropenes and sesquiterpenes caused a significant reduction on *S. littoralis* larval growth after 3, 6 and 9 days of feeding in treated diet with 500, 1000 and 2000 mg/kg (Table 2). Remarkable growth inhibition (GI) was observed after three days of treatment as the larval growth inhibition (GI) ranged between 45.4 and 100%. Cuminaldehyde (GI = 96.2%) and 1,8-cineole (GI = 95.9%) exhibited the strongest larval growth inhibition at 500 mg/kg. Both compounds caused higher growth inhibition than pyriproxyfen at this concentration. At 1000 mg/kg, 1,8-cineole (GI = 100.0%), eugenol (GI = 86.2%) and cuminaldehyde (GI = 86.0%) were the most potent growth inhibitors, while 1,8-cineole (GI = 100.0%) and cuminaldehyde (GI = 85.2%) had the highest growth inhibition at 2000 mg/kg. After 6 days of treatment, 1,8-cineole revealed the highest growth inhibition at the three tested concentrations with 82.8, 86.9 and 91.5% growth inhibition at 500, 1000 and 2000 mg/kg, respectively. After 9 days treatment, 1,8-cineole was the most potent growth inhibitor among the compounds at the three tested concentrations with growth inhibition percentages of 87.9, 73.5 and 87.4 at 500, 1000 and 2000 mg/kg, respectively. The other compounds showed growth inhibition ranged between 29.3 and 79.2%.

Insecticidal activity of monoterpenes, phenylpropenes and sesquiterpenes

The toxicity of monoterpenes, phenylpropenes and sesquiterpenes on the 2nd larval instar of *S. littoralis* after feeding on treated semi-artificial diet for 3, 6 and 9 days is presented in Table 3. The results showed that the mortality percentages

Table 1 Antifeedant activity of monoterpenes, phenylpropenes and sesquiterpenes on the 2nd instar larvae of *Spodoptera littoralis* after 3, 6 and 9 days of feeding on treated semi-artificial diet[a]

Compound	Antifeedant index (% ± SE)[b]								
	After 3 days			After 6 days			After 9 days		
	500 mg/kg	1000 mg/kg	2000 mg/kg	500 mg/kg	1000 mg/kg	2000 mg/kg	500 mg/kg	1000 mg/kg	2000 mg/kg
Control	0.0 ± 0.00f	0.0 ± 0.00f	0.0 ± 0.00f	0.0 ± 0.00f	0.0 ± 0.00d	0.0 ± 0.00 g	0.0 ± 0.00 h	0.0 ± 0.00e	0.0 ± 0.00 g
(−)-Carvone	20.4 ± 1.85bc	27.8 ± 3.21bcd	-[c]	37.9 ± 1.61abcd	30.7 ± 5.65c	–	55.2 ± 1.48ef	47.2 ± 1.99d	–
1,8-Cineole	1.9 ± 4.91ef	13.0 ± 1.85def	18.5 ± 1.85 cd	33.9 ± 0.81bcde	56.5 ± 1.40a	78.2 ± 2.46a	73.0 ± 1.12b	61.3 ± 1.84c	51.3 ± 2.44ef
trans-Cinnamaldehyde	33.3 ± 3.21a	44.4 ± 5.56a	44.4 ± 0.64a	44.4 ± 5.04abc	37.1 ± 2.80bc	29.0 ± 3.14de	83.5 ± 2.32a	66.4 ± 3.35bc	80.1 ± 1.22a
(−)-Citronellal	16.7 ± 3.21 cd	11.1 ± 6.42ef	25.9 ± 3.71b	23.4 ± 2.91e	35.5 ± 0.81c	21.8 ± 2.14e	65.0 ± 1.84 cd	73.7 ± 1.84ab	77.6 ± 0.49ab
Cuminaldehyde	31.5 ± 4.91a	18.5 ± 4.91 cde	25.9 ± 3.71b	1.7 ± 0.27f	39.5 ± 4.20bc	9.5 ± 2.10f	44.0 ± 1.29 g	61.6 ± 4.25c	53.5 ± 2.32e
P-Cymene	29.6 ± 1.85ab	29.6 ± 3.71abc	29.5 ± 3.71b	47.6 ± 0.81a	33.1 ± 2.14c	30.6 ± 2.14cde	49.6 ± 3.60 fg	58.2 ± 2.58c	46.5 ± 1.71f
Eugenol	11.1 ± 5.56cdef	16.7 ± 0.00 cde	14.8 ± 1.96de	38.7 ± 4.91abcd	49.2 ± 3.70ab	23.4 ± 5.65e	63.3 ± 1.22 cd	47.9 ± 1.48d	47.0 ± 4.55f
Farnesol	13.0 ± 6.68cde	37.0 ± 12.98ab	14.8 ± 1.34de	32.3 ± 5.04cde	29.0 ± 4.27c	39.5 ± 4.91 cd	65.9 ± 2.44c	62.8 ± 2.11c	71.8 ± 1.95b
(*Z*,*E*)-Nerolidol	11.1 ± 3.21 cdef	18.5 ± 3.71 cde	25.9 ± 1.96b	34.7 ± 5.04bcde	32.3 ± 6.41c	34.7 ± 3.70 cd	59.1 ± 2.95de	64.5 ± 3.17c	61.1 ± 1.99 cd
(−)α-Pinene	18.5 ± 3.71 cd	18.5 ± 1.85 cde	9.3 ± 1.85e	28.2 ± 4.91de	38.7 ± 2.14bc	39.5 ± 3.40 cd	55.0 ± 1.76ef	58.6 ± 3.78c	56.0 ± 2.81de
α-Terpinene	20.4 ± 1.85bc	13.0 ± 1.85def	9.3 ± 1.85e	50.0 ± 0.81a	41.9 ± 3.70bc	40.3 ± 2.90c	55.7 ± 2.12ef	61.1 ± 2.99c	62.8 ± 1.52c
Pyriproxyfen	7.4 ± 1.85def	7.4 ± 1.85ef	11.5 ± 1.85de	45.2 ± 7.70ab	30.7 ± 6.61c	51.6 ± 3.70b	76.9 ± 1.22b	77.1 ± 0.97a	80.5 ± 1.71a

[a] Data are expressed as mean values ± SE from experiments with five replicates of 10 larvae each

[b] Mean values within a column sharing the same letter are not significantly different at the 0.05 probability level

[c] Complete mortality

Table 2 Growth inhibitory effect of monoterpenes, phenylpropenes and sesquiterpenes on the 2nd instar larvae of *Spodoptera littoralis* after 3, 6 and 9 days of feeding on treated semi-artificial diet[a]

Compound	Growth inhibition (% ± SE)[b]								
	After 3 days			After 6 days			After 9 days		
	500 mg/kg	1000 mg/kg	2000 mg/kg	500 mg/kg	1000 mg/kg	2000 mg/kg	500 mg/kg	1000 mg/kg	2000 mg/kg
Control	0.0 ± 0.00f	0.0 ± .00f0	0.0 ± 0.00 g	0.0 ± 0.00i	0.0 ± 0.00i	0.00 ± 0.00i	0.0 ± 0.00 k	0.0 ± 0.00 k	0.0 ± 0.00 h
(−)-Carvone	67.4 ± 0.79c	83.3 ± 1.73bc	- [c]	40.2 ± 1.64 fg	61.1 ± 2.32d	–	41.2 ± 1.85 g	35.0 ± 2.08 h	–
1,8-Cineole	95.9 ± 0.60a	100.0 ± 0.00a	100.0 ± 0.00a	82.8 ± 2.00b	86.9 ± 1.02b	91.5 ± 0.26b	87.9 ± 0.56b	73.5 ± 0.98b	87.4 ± 0.53b
trans-Cinnamaldehyde	57.7 ± 2.27e	69.4 ± 1.04d	59.8 ± 0.17e	68.6 ± 0.31c	45.7 ± 1.81ef	53.8 ± 4.01ef	46.7 ± 1.57ef	47.0 ± 1.05ef	79.2 ± 0.54c
(−)-Citronellal	68.2 ± 1.73c	80.3 ± 0.60c	64.4 ± 0.35ef	52.4 ± 4.47e	49.8 ± 0.63ef	64.0 ± 2.00d	56.7 ± 1.96d	60.9 ± 2.12c	78.6 ± 0.94c
Cuminaldehyde	96.2 ± 0.46a	86.0 ± 0.79b	85.2 ± 0.17b	43.9 ± 1.38f	77.5 ± 1.60c	75.6 ± 1.21c	30.4 ± 1.56i	69.9 ± 1.41b	64.2 ± 1.27d
P-Cymene	78.8 ± 1.87b	73.9 ± 2.68d	76.3 ± 1.21c	61.1 ± 1.92d	63.9 ± 3.03d	61.2 ± 2.38d	37.1 ± 1.45 h	53.3 ± 1.57d	48.9 ± 1.57e
Eugenol	65.5 ± 2.7 cd	86.2 ± 1.73b	72.4 ± 0.69 cd	55.9 ± 4.43de	51.0 ± 0.84ef	53.6 ± 1.55ef	42.0 ± 1.69 g	29.3 ± 2.16i	36.5 ± 1.82 g
Farnesol	58.9 ± 0.69e	45.4 ± 0.91e	53.2 ± 3.98f	33.4 ± 3.94gh	38.1 ± 4.47 g	21.4 ± 4.96 g	50.3 ± 0.79e	43.5 ± 1.45 g	61.8 ± 0.83d
(*Z*,*E*)-Nerolidol	57.3 ± 0.46e	71.0 ± 3.68d	82.0 ± 1.38b	30.5 ± 2.43 h	44.0 ± 4.30 fg	48.2 ± 3.11f	61.8 ± 1.75c	48.9 ± 1.60de	49.8 ± 1.75e
(−)α-Pinene	59.9 ± 5.09de	70.5 ± 1.31d	70.5 ± 2.07 cd	33.2 ± 3.45 gh	52.6 ± 0.80e	53.2 ± 1.16ef	43.0 ± 1.61 fg	43.1 ± 2.44 fg	41.6 ± 1.71f
α-Terpinene	75.8 ± 1.70b	79.5 ± 3.37c	70.3 ± 3.46d	57.5 ± 1.28de	66.0 ± 2.14d	59.3 ± 2.27de	47.6 ± 0.95e	42.5 ± 2.37 fg	36.0 ± 2.41 g
Pyriproxyfen	79.6 ± 1.83b	96.2 ± 0.69a	100.0 ± 0.00a	90.7 ± 0.16a	97.1 ± 0.22a	99.9 ± 0.07a	93.2 ± 0.15a	98.0 ± 0.18a	99.1 ± 0.13a

[a] Data are expressed as mean values ± SE from experiments with five replicates of 10 larvae each

[b] Mean values within a column sharing the same letter are not significantly different at the 0.05 probability level

[c] Complete mortality

Table 3 Toxicity of monoterpenes, phenylpropenes and sesquiterpenes on 2nd instar larvae of *Spodoptera littoralis* after 3, 6 and 9 days of feeding on treated semi-artificial diet[a]

Compound	Mortality (% ± SE)[b]								
	After 3 days			After 6 days			After 9 days		
	500 mg/kg	1000 mg/kg	2000 mg/kg	500 mg/kg	1000 mg/kg	2000 mg/kg	500 mg/kg	1000 mg/kg	2000 mg/kg
Control	0.0 ± 0.00c	0.0 ± 0.00f	0.0 ± 0.00f	0.0 ± 0.00c	0.0 ± 0.00f	0.0 ± 0.00 h	0.0 ± 0.00f	0.0 ± 0.00f	0.0 ± 0.00 h
(−)-Carvone	13.0 ± 3.34c	33.0 ± 3.34 cd	100.0 ± 0.00a	13.0 ± 3.34c	33.0 ± 3.34bc	100.0 ± 0.00a	13.0 ± 3.34def	37.0 ± 3.34bc	100.0 ± 0.00a
1,8-Cineole	7.0 ± 3.34c	67.0 ± 3.34a	97.0 ± 3.34a	10.0 ± 5.78c	80.0 ± 5.78a	97.0 ± 3.34a	13.0 ± 3.34def	80.0 ± 5.78a	97.0 ± 3.34a
trans-Cinnamaldehyde	7.0 ± 3.34c	0.0 ± 0.00f	17.0 ± 3.34ef	7.0 ± 3.34	10.0 ± 3.34ef	17.0 ± 3.34 fg	40.0 ± 5.78b	13.0 ± 3.34ef	20.0 ± 5.78 fg
(−)-Citronellal	20.0 ± 5.78c	13.0 ± 3.34ef	43.0 ± 6.67d	20.0 ± 5.78bc	20.0 ± 5.78cde	47.0 ± 3.34 cd	20.0 ± 3.34 cde	23.0 ± 3.34 cde	60.0 ± 5.78c
Cuminaldehyde	50.0 ± 5.78a	47.0 ± 3.34b	77.0 ± 3.34b	60.0 ± 5.78a	47.0 ± 3.34b	77.0 ± 3.34b	67.0 ± 3.34a	47.0 ± 3.34b	77.0 ± 3.34b
P-Cymene	33.0 ± 3.34b	30.0 ± 0.00 cd	30.0 ± 5.78de	33.0 ± 3.34b	30.0 ± 0.00bcd	30.0 ± 5.78ef	33.0 ± 3.34bc	33.0 ± 3.34bcd	30.0 ± 5.78ef
Eugenol	7.0 ± 3.34c	40.0 ± 5.78bc	60.0 ± 5.78c	7.0 ± 3.34c	43.0 ± 3.34b	40.0 ± 5.78de	10.0 ± 0.00ef	70.0 ± 5.78a	40.0 ± 5.78de
Farnesol	0.0 ± 0.00c	13.0 ± 3.34ef	3.0 ± 3.34f	0.0 ± 0.00c	13.0 ± 3.34def	10.0 ± 0.00 gh	0.0 0.0 ± 0.00ef	13.0 ± 3.34ef	20.0 ± 0.00 fg
(*Z*,*E*)-Nerolidol	13.0 ± 3.34c	7.0 ± 3.34f	7.0 ± 3.34f	13.0 ± 3.34c	13.0 ± 3.34def	7.0 ± 3.34gh	13.0 ± 3.34def	13.0 ± 3.34ef	7.0 ± 3.34gh
(−)α-Pinene	10.0 ± 5.78c	13.0 ± 3.3ef	13.0 ± 3.34ef	10.0 ± 5.78c	20.0 ± 5.78cde	20.0 ± 5.78 fg	13.0 ± 0.00def	20.0 ± 5.78de	20.0 ± 5.78 fg
α-Terpinene	10.0 ± 5.78c	10.0 ± 5.8ef	10.0 ± 5.78f	13.0 ± 3.34c	13.0 ± 3.34def	10.0 ± 5.78gh	17.0 ± 3.34def	13.0 ± 3.34ef	10.0 ± 0.00 fgh
Pyriproxyfen	13.0 ± 3.34c	23.0 ± 3.3de	30.0 ± 5.78de	20.0 ± 5.78bc	47.0 ± 3.34b	57.0 ± 3.34c	30.0 ± 5.78bcd	47.0 ± 3.34b	57.0 ± 3.34d

[a] Data are expressed as mean values ± SE from experiments with five replicates of 10 larvae each

[b] Mean values within a column sharing the same letter are not significantly different at the 0.05 probability level

enhanced by increasing the concentration and exposure period. Cuminaldehyde was the most potent compound at concentration of 500 mg/kg, while1,8-cineole and (−)-carvone revealed the highest insecticidal activity at concentrations of 1000 and 2000 mg/kg, respectively, after the three exposure times. At 2000 mg/kg, (−)-carvone induced complete larval mortality (100%) and 1,8-cineole caused 97.0% mortality after the three exposure times. Cuminaldehyde, 1,8-cineole and (−)-carvone showed higher toxicity than pyriproxifen. In contrary, farnesol, nerolidol, α-pinene and α-perpinene revealed weak insecticidal activity at the three tested concentrations.

Inhibitory effect of phenylpropenes and sesquiterpenes on α-amylase and total proteases

Among the tested compounds, *trans*-cinnamaldehyde, eugenol and farnesol caused high antifeedant and growth inhibition activities. Thus, these three compounds were tested for their inhibitory effects on α-amylase and total proteases isolated from *S. littoralis* larvae. The tested compounds revealed remarkable inhibitory effect on α-amylase activity (Table 4). *trans*-Cinnamaldehyde caused the highest inhibitory activity among the tested compound with IC_{50} value of 0.03 mM, followed by farnesol with IC_{50} values of 0.04 mM, then eugenol with IC_{50} value of 0.06 mM. On the other hand, eugenol (IC_{50} = of 0.24 mM) caused the greatest inhibition of the total proteases activity, followed by *trans*-cinnamaldehyde (IC_{50} = 1.12 mM) and farnesol (IC_{50} = 2.33 mM).

Discussion

Plants have numerous secondary metabolites that possess plant protection properties against different pests and pathogens. Application of these natural products in insect pest management programs has received much attention in recent years due to drawbacks associated with unwise use of synthetic insecticides. Particularly, the plant compounds with feeding deterrent and growth inhibitory properties gained more attention in IPM programs because these compounds are important mediators of plant–insect interactions and insect behavior manipulators. The feeding deterrent and growth regulatory properties of plant extracts and secondary metabolites have been studied against lepidopteran larvae and other insects (Ballesta-Acosta et al. 2008; Rani and Murty 2009; Zapata et al. 2009; Pavela 2010).

In the current study, monoterpenes, phenylpropenes and sesquiterpenes revealed pronounced antifeeding activity on the second larval instar of *S. littoralis*. Among the tested compounds, *trans*-cinnamaldehyde, 1,8-cineole, (−)-citronellal and farnesol were the most effective antifeedant, particularly after 9 days of treatment. The results indicated that the antifeedant activity of tested compounds was time dependent. For example, *trans*-cinnamaldehyde and *p*-cymene were relatively the most active antifeedants after 3 days of treatment, while 1,8-cineole and α-terpinene revealed the highest antifeedant activity after 6 days of treatment. Additionally, *trans*-cinnamaldehyde and (−)-citronellal were the most effective antifeedants after 9 days of treatment. The quick antifeedant activity (after 3 days) of compounds may be attributed to the repellent effect of these compounds or to their unacceptable taste for the larvae. So the larvae did not eat at the beginning but after starvation the larvae started feeding. The late antifeedant activity (after 6 and 9 days) of compounds may be due to the inhibitory effect of compounds on digestive enzymes which allowed larvae to eat at the beginning and stop eating later on. The enhancing of antifeedant activity of all compounds with increasing exposure time could be attributed to the combine effect of repellent, unacceptable taste and inhibition of digestive enzymes.

In the literature, there are no recorded studies on the antifeeding activity of tested compounds on *S. littoralis* larvae. However, essential oils whose major constituents are

Table 4 Inhibitory effect of phenylpropenes and sesquiterpenes on the activity of *Spodoptera littoralis* larval α-amylase and total proteases

Enzyme	Compound	IC_{50} [a] (mM) (Confidence limits)	Slope ± SE [b]	Intercept ± SE [c]	(X^2) [d]
α-Amylase					
	trans-Cinnamaldehyde	0.03 (0.02–0.05)	1.98 ± 0.15	−3.04 ± 0.26	11.43
	Eugenol	0.06 (0.05–0.08)	1.24 ± 0.09	−2.25 ± 0.18	1.06
	Farnesol	0.04 (0.035–0.051)	1.48 ± 0.12	−2.42 ± 0.21	1.40
Proteases					
	trans-Cinnamaldehyde	1.12 (0.91–1.37)	1.27 ± 0.11	−0.06 ± 0.06	3.49
	Eugenol	0.24 (0.20–0.30)	1.46 ± 0.14	3.49 ± 0.33-	2.10
	Farnesol	2.33 (1.89–2.84)	2.29 ± 0.11	−0.47 ± 0.08	2.92

[a] The concentration causing 50% enzyme inhibition

[b] Slope of the concentration-inhibition regression line

[c] Intercept of the regression line ± SE

[d] Chi square value

monoterpenes and sesquiterpenes have been shown to have antifeedant activity against the fourth larval instar of *S. littoralis* (Ali et al. 2017). Similarly, Gonzalez et al. (1997) explained the antifeedant activity of sesquiterpenes isolated from seven Celastraceae species against fifth larval instar of *S. littoralis*. Other phytochemicals, such as diterpenes (eriocephalin, salviacoccin, aethiopinone and oxocandesalvone), coumarins (oxypeucedanin, xanthotoxin, isoimperatorin and prangol), drimanes (drimendiol, isodrimeninol, isotadeonal and polygodial) and limonoids (khayalactol, khayanolide D, 2-hydroxyseneganolide, 1-*O*-acetylkhayanolide A, khayanolide A and methyl angolensate) have been described to show significant antifeedant activity against larval instars of this insect (Ballesta-Acosta et al. 2008; Zapata et al. 2009). On the other hand, some of the tested compounds have been shown to possess antifeedant activity against other insects. For example, eugenol and 1,8-cineole revealed high feeding-deterrence effect on *Rhyzopertha dominica* and *Tribolium castaneum* (Ukeh and Umoetok 2011). Other monoterpenes, such as thymol, (+)-limonene and (±)-camphene had antifeedant activity against *T. castaneum*, *R. dominica* and *Solanum tuberosum* (Szczepanik et al. 2009; Kanda et al. 2016).

The results indicated that the tested compounds are promising growth inhibitors of second larval instar of *S. littoralis* for the first time. These results are supported by earlier studies in which some sesquiterpenes, such as drimendiol, isodrimeninol were reported to cause growth inhibition of *S. littoralis* larvae (Zapata et al. 2009). It is also documented that eudesmane sesquiterpenes inhibited the growth of *S. frugiperda*, (Sosa et al. 2017). Moreover, some triterpenes caused growth inhibition of *S. littoralis* larvae, such as limonoids from *Khaya senegalensis*, *Chukrasia tabularis* and *Swietenia mahogani* (El-Aswad et al. 2003; Abdelgaleil and El-Aswad 2005). The potent growth inhibitory effects observed here indicated that the tested monoterpens, phenylpropenes and sesquiterpenes could strongly delay the development of the insect, produce smaller pupa, reduce adult emergence and also decrease the fecundity and fertility of the emerged females which led to drastic reduction in insect population as well as make insects more susceptible to diseases and other control methods. Therefore, these compounds could be used as promising candidates in the plant protection programs of this insect.

Besides their effects on feeding-deterrence and growth inhibition, the tested compounds also showed insecticidal activity against of *S. littoralis* larvae. However, the majority of compounds showed less toxic effect than growth inhibition and antifeedant effects. The results also showed that (−)-carvone and 1,8-cineole are the most active toxicants against the 2nd larval instar of *S. littoralis*. This finding is supported by earlier studies in which (−)-carvone and 1,8-cineole showed strong fumigant and contact toxicities against the third larval instar of this insect (Abdelgaleil 2010). The toxicity of phenylpropenes and monoterpenes observed in the present study is also supported by earlier studies in which *trans*-ethyl cinnamate, thymol, carvacrol, trans-anethole and piperitone revealed contact toxicity against the third larval instar (Abdelgaleil et al. 2008; Pavela 2014), and γ-terpinene and terpinen-4-ol caused contact toxicity against the fourth larval instar of *S. littoralis* (Abdelgaleil et al. 2008; Abbassy et al. 2009). Moreover, few sesquiterpenes have been reported to cause residual toxicity against *S. littoralis* larvae (Srivastav et al. 1990; Gonzalez et al. 1997).

It has been suggested that the antifeedant compounds deter insect feeding via sensory perception, such as having an unpalatable taste to insects and/or via postingestive effects (Abdelgaleil and El-Sabrout 2018). Essential oils and their major constituents (monoterpenes, phenylpropenes and sesquiterpenes) possess aromatic properties and cause insects disgusted by food and reduce or stop feeding (Arasu et al. 2013). Some essential oils and monoterpenes have been reported to inhibit α-amylase and other digestive enzymes (Basak and Candan 2010; Sudha et al. 2011; Kohl et al. 2015). The results of this study supported the later hypothesis as the tested compounds induced inhibitory effects on α-amylase and total proteases activities. In fact, other plant secondary metabolites, such as terpenes (squalene, lupeol, oleanoic acid, ursolic acid and betulinic acid) have been shown to inhibit α-amylase activity (de Sales et al. 2012).

Although the effect of monoterpenes, phenylpropenes and sesquiterpenes on total proteases were not previously reported some terpenoids, such as diterpenes and triterpenes and have been shown to inhibit digestive proteases of Colorado potato beetle, *Leptinotarsa decemlineata* (Ortego et al. 1999). Furthermore, azadirachtin, tetranortriterpene, has been reported to inhibit the activity of digestive proteases in larvae of *S. litura* which indicated its disruption effects on the digestive process in insects (Koul et al. 1996), *Manduca sexta* (Timmins and Reynolds 1992) and *S. littoralis* (Abou-Taleb 2016).

In conclusion, the tested monoterpenes, phenylpropenes and sesquiterpenes caused interesting antifeedant and growth inhibitory effects as well as induced toxicity of *S. littoralis* larvae. Therefore, these compounds, particularly, *trans*-cinnamaldehyde, cuminaldehyde, 1,8-cineole, (−)-carvone and eugenol, could be serve as effective bio-insecticides for managing this polyphagous insect. However, studies on their binary mixtures and their mixtures with synergists are recommended to enhance their efficacy against target insect. Further studies are also needed on formulations and stability of these compounds under field condition. Also, the efficacy of the formulations on target and non-target organisms should be addressed before commercial use.

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Compliance with ethical standards

Conflict of Interest The authors declare that they have no conflict of interest.

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