



Biological activity of tannins extracts from processed *Camellia sinensis* (black and green tea), *Vicia faba* and *Urtica dioica* and *Allium cepa* essential oil on three economic insects

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Received: 9 February 2022 / Accepted: 20 October 2022 / Published online: 10 November 2022
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Abstract

Nowadays to move toward a more sustainable agriculture, use of pesticide alternatives that have positive effects and play critical roles toward reducing the use of pesticides should be used. Laboratory bioassays were carried out to analyze the bioactivity of tannins isolated from urtica weed leaves (U), bean hull (B), black tea (BT) and green tea (GT) against larvae of cotton leafworm *Spodoptera littoralis* (Boisduval) using food mixing technique and essential oil isolated from onion against larvae of cotton leafworm, adults of rice weevil and houseflies using fumigation technique. The results showed that during the first week of treatment, tannins extracted from U had the strongest antifeedant activity with an EC_{50} of 33.034 $\mu\text{g/g}$ followed by tannins extracted from B (EC_{50} = 47.839 $\mu\text{g/g}$). In the second and third week, tannins isolated from B depicted highest antifeedant activity (EC_{50} = 37.733 and 84.828, respectively). Furthermore, the isolated tannins induced notable larval growth inhibition on *S. littoralis*. On the other hand, mortality percentage of onion essential oil on tested insects clearly increased with both increased concentration and exposure time. The LC_{50} ($\mu\text{g/cm}^3$) at 30 min reflected that the essential oil had a greater toxicity to cotton leafworm with a LC_{50} = 2.15 $\mu\text{g/cm}^3$ while least toxic to house flies (LC_{50} = 16.09). The repellency action based on LT_{50} values was seen to be highly effective in houseflies (LT_{50} = 1.85 min). The results demonstrated that tannins and essential oil could be applicable in the management of insect pests to decrease ecologically detrimental effects of synthetic insecticides.

Keywords Tannins · Essential oil · Insects · Antifeedant · Growth inhibitory · Repellence

Introduction

Environmental contamination, as well as the emergence of pest that are resistant to synthetic pesticides have caused a great deal of concern, prompting the search for new, safer alternatives. Hence, it is essential to develop alternative

pesticides that are effective and environmentally friendly (Caicedo-López et al. 2021; Chacón et al. 2021).

Tannins are generally complex chemical substances derived from phenolic acids and are naturally occurring polyphenol compounds present in the plant kingdom (Smeriglio et al. 2017). Tannins can be grouped into two classes, i.e., hydrolysable and non-hydrolysable tannins. Hydrolysable tannins have ester or glucosidal bonds easily broken down by acids, whereas non-hydrolysable tannins have benzene nuclei (Ogwuru and Adamczeski 2000). The final products of tea leaves are usually a result of polyphenol oxidation which is found naturally abundant in them. Black tea (BT) is produced by complete oxidation (fermentation) while green tea (GT) production bypasses oxidation step (Khasnabis et al. 2015). Tannins have a powerful detrimental effect to phytophagous insects. They alter the growth and development of insects by binding to the proteins, decreasing the ability to absorb nutrients and by inducing midgut lesions (Dubey et al. 2016; Martemyanov et al. 2006). Tannins are

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mouth-puckering bitter polyphenols and are effective feeding deterrents to many insect pests (Dubey et al. 2016). Solid–liquid extraction is dedicated to the extraction of tannins exclusively based on contact between a solvent and a solid substance, without any other mechanism of assistance (de Hoyos-Martinez et al. 2019). The extraction is typically carried out using a Soxhlet apparatus, experimental extraction procedure of which has been presented in several works (Cuong et al. 2020; De Castro and Priego-Capote 2010). The effectiveness of Soxhlet extraction is primarily based on the solvent's evaporation temperature and polarity (Markom et al. 2007). The solvent like ethanol water mixture is used for the extraction and delivers tannins from herbal plants with high extract yield (Kamarudin et al. 2016). Tannins are also present in many dry botanical products such as bean hull (B), BT and GT and fresh plant such as leaves of *Urtica* plant. Nettle (*Urtica dioica* L.) which is a perennial wild plant of the Urticaceae family, genus *Urtica*, has long been used in the cosmetic, food and pharmaceutical industries, as all parts (leaves, stalks and roots) show a rich composition of bioactive compounds (Kregiel et al. 2018) as well as, vitamins, minerals, polyphenols such as phenolic acids, flavonoids and pigments (Repajić et al. 2020).

Essential oils are generally concentrated hydrophobic liquid having volatile chemicals compound derived from aromatic plants (Svoboda and Hampson 1999). A wide spectrum of activity against insects and plant pathogenic fungi have been demonstrated (Regnault-Roger et al. 2012). Essential oils are easily obtained by steam distillation of plant material and contain several volatile terpenes and phenolic of low-molecular-weight. Essential oils from onions have in the past been traditionally used for treatment and prevention of many diseases and disorders (Keusgen 2002).

It is a truism that agriculture has a pivotal role to play to encounter the growing demands for food. However, a greater percentage of the world's agricultural commercial produces are lost to insects and other pest infestations (UN-FAO 2019). Pesticides have been used to protect plants during production, storage and transportation although, its environmental impact has received increasing attention due to its alarming human health concerns and its impact on water quality and agricultural products (Gavrilescu 2005). In recent years, several attempts to develop new insecticides have been spotlighted on tannins and essential oil. This could be due to their potency effectiveness on insects and low toxicity on nontarget organisms (Lengai et al. 2020).

Cotton leafworm *Spodoptera littoralis* (Boisduval), Rice weevil *Sitophilus oryzae* (Linnaeus) and housefly *Musca domestica* (Linnaeus) are of great concerns to food crops, stored product and public health, respectively. Synthetic pesticides have widely been used to control these insects (Benhalima et al. 2004; El Sherif et al. 2022; Hawkins et al. 2019). However, in general, due to the resistance and

toxicity impacts of the synthetic insecticides, there is need to develop effective and low risks alternatives (Chowański et al. 2014). Among the alternatives, antifeedant activity of botanical sources which based on chemical composition (Adeyemi and Mohammed 2014; Bruno et al. 2002) and inhibitory effect of plant tannins based on its concentration (Nomura and Itioka 2002).

Therefore, this research aimed to assess the potency and repellence ability of onion essential oil on cotton leafworm, rice weevil and houseflies as well as the growth inhibitory and antifeedant effects of tannins on cotton leafworm.

Material and methods

Plant materials

The nettle plant *Urtica dioica* (Linnaeus) which grows in cotton crop was identified by the botanists at the Plant pathology department, Faculty of Agriculture, Alexandria University, Egypt. The fresh leaves of *Urtica* weeds and bulbs of red onions *Allium cepa* (Linnaeus) (Variety Beheri) were collected in June 2020 from Research station, Abbis farm, Alexandria, Egypt. Bean seeds, Black tea and Green tea commodities were also bought from Carrefour market in Alexandria, Egypt.

Test insect

Laboratory cotton leafworm *S. littoralis* and rice weevil *S. oryzae* strains were donated by the Bioassay Laboratory, Pesticide Chemistry Department, Faculty of Agriculture, Alexandria University. The cotton leafworm *S. littoralis* colony was reared on semi-artificial diet under laboratory conditions (El-Minshawy and Zeid 2009). The colony of rice weevil *S. oryzae* was reared on wheat kernels under laboratory conditions of 27 °C and 65% relative humidity (Athanasios et al. 2017). Adult house flies *Musca domestica* were obtained from the animal husbandry yard in Abbis farm, Alexandria, Egypt, restrained in a wire netting cage and fed with a mixture of honey and powdered milk shortly before the experiment (Vignau et al. 2003).

Extraction and characterization of tannins from different samples

Preparation of samples

The black tea and green tea were finely powdered. The bean seeds were soaked in water to germinate for 48 h. The crust of seeds was separated and dried for two days at room temperature, after that, crushed, milled in a knife mill (2 mm

screen) to obtain 150 g powder and subsequently stored in a glass bottle at room temperature.

Fresh leaves of *Urtica dioica* were washed with distilled water, cut into small pieces 2 cm² then dried at room temperature for two days, subsequently, crushed, milled in a knife mill (2 mm screen) to obtain 150 g of powder and afterward stored in glass bottles at room temperature.

Extraction of tannins

The different samples (100 g for each) were extracted in a Soxhlet apparatus (Cuong et al. 2020; de Hoyos-Martinez et al. 2019) with petroleum ether (60–40) for 15 min followed by benzene for 15 min to remove fatty acids and dyes to become colorless. After removing two solvents, the samples were extracted with ethanol 80% for 24 h (Kamarudin et al. 2016). Extracts were transported to a tarred, round-bottomed flask and concentrated under vacuum using rotary evaporator to form a thick extract. In a vacuum oven at 60 °C, the sample extracts were then dried until a solid material was provided. The freshly prepared tannin is nearly a colorless amorphous powder which gradually oxidizes in the air, to a black brown mass. The amount of extract was determined by weight difference and kept in the dark at 4 °C until used/further analysis.

Characterization of tannins

Determination of total tannin content

Total content of tannins was determined using the Folin–Ciocalteu reagent as reported by Kyamuhangire et al. (2006). Briefly, 2 mL of Folin reagent (diluted 10 times) was added to 1 mL of aqueous solution 10% of each ethanol extract. Two mL of sodium carbonate (75 g L⁻¹) was then added. The mixture was incubated in a water bath at 50 °C for 15 min and the absorbance was read at 700 nm. All measurements were taken in triplicates. Standard solutions of tannic acid were used for quantification. The results were expressed as milligram of tannic acid equivalent (TAE) per gram of air-dry sample (mg TAE/g sample) and as milligram of TAE per gram of dry extract (mg TAE/g extract).

Determination of condensed tannins

The condensed tannins content in such obtained extracts was determined using vanillin (Price et al. 1978) with some modifications. One milliliter of water solution of each extract (10%) was mixed with 5 mL of vanillin reagent, containing 0.5% w/v vanillin and 8% v/v concentrated HCl in acetic acid. Then, samples and controls (without vanillin) could hold for 20 min in darkness and afterward absorbance was read at 500 nm. The content of condensed tannin was shown

as absorbance units per 1 mg of tannin fraction (Karamac et al. 2007).

Determination of hydrolysable tannins

The content of hydrolysable tannins was determined using potassium iodate as reported by Bossu et al. (2006) method. Five milliliters of KIO₃ aqueous solution (2.5% v/v) were heated for 7 min at 30 °C, then 1 mL of the aqueous solution of each extract (10%) was added. The mixture was placed in a water bath for 2 min at 30 °C then the absorbance at 550 nm was read. The results of hydrolysable tannins were obtained as absorbance units per 1 mg tannin fraction.

Isolation of the onion essential oil

Studies have shown that essentials oil from onions can be effectively extracted by hydro-distillation using Clevenger-type apparatus as reported by Sharma and Tripathi (2006) and Mnayer et al. (2014).

Two kilograms of onion bulbs which were free of external damages were selected and chopped manually. Twice, 500 g of the onion pulps was cut into small pieces and subjected to hydro-distillation in a Clevenger-type apparatus with 700 mL distilled water to obtain the EOs (Clevenger 1928). The extraction process continued until no more EO was obtained (about 4 h). The EO was collected, dried over anhydrous sodium sulfate to remove all traces of water and stored in a refrigerator at −4 °C away from light until further analysis. The onion EO was analyzed by gas chromatography–mass spectrometry (GC–MS) to identify the major chemical components.

GC/MS analysis of essential oil

The essential oil composition was analyzed using gas chromatography/mass spectrometry (GC/MS) with the following specifications: A Trace GC Ultra/Mass Spectrophotometer ISQ (Thermo Scientific) instrument equipped with flame ionization detectors (FID) and TG1MS column. It was used as carrier gas with a linear flow velocity of 50 cm/s (flow rate of 1 mL/min), and the oven temperature was held at 30 °C for 1 min then increased from 30 °C to 180 °C (10 °C/min) and 180–200 °C (10 °C/min) with post run (off) at 350 °C. The GC/MS was equipped with a ZB-5MS Zebron capillary column (length 30 m × 0.25 mm ID, 0.25 µm film thickness; Agilent). Sample (1 µL) was injected at 250 °C, with split/split-less injector (50:1 split ratio) in the split-less mode flow with 11 mL/min. The mass spectrometer was scanned from 50–500 m/z at five scans per second. Scan time: 0.2 s; mass range: 50 to 550 amu. The separated components were subsequently identified by their retention indices (RI) relative to n-alkanes (C6–C20), and by matching

their mass spectra to the NIST, WILEY library database (> 90% match) under identical GC–MS conditions (Adams 2007; Davies 1990; Van Den Dool and Kratz 1963). Peak area percent was used for obtaining quantitative data of the components with the Xcalibur 2.0 software (Agilent Technologies). Peak quantitation was performed using Met-Idea program (Broeckling et al. 2006).

Fumigant and repellent activity of essential oil

Fumigant activity

The fumigant toxicity of the essential oil isolated from onions was examined on adult house fly, adult rice weevil and cotton leafworm larvae. Glass jars (500 ml, 10 cm height and 8 cm diameter) were used as a fumigation chambers. Initially, medium sized round filter papers of diameter 5.5 cm were attached to the under surface of the glass jars' screw caps. Essential oil Solutions in acetone were prepared at concentrations corresponding to 1, 2.5, 5, 7.5, 10, 15, 25 and 50 µg/cm³ as well as acetone alone as control. These concentrations were applied on the filter paper disks. Ten insects were introduced in each jar and the caps were screwed tightly to the lead of a glass jar using a seal tape. Three replicates of each control and treatment were measured. The percentage of mortality of each concentration was recorded at different time intervals and expressed as µg/cm³ volume of space (Broussalis et al. 1999). The mortality percentages were calculated and corrected according to Abbott equation (Abbott 1925). Accordingly, LC₅₀ and LT₅₀ values were determined.

Repellent activity

Repellent activity of the essential onion oil on adult houseflies, rice weevil and cotton leafworm larvae (20 insects each) was carried out using WHO standard tube. The degree of repellence can be observed by the aggregation of insects in the separate portions of the WHO tube with time. It is important to note that each experiment was repeated three times (Procópio et al. 2003). Consequently, RT₅₀ was determined.

Antifeedant activity and growth inhibitory of tannins

The antifeedant activity and growth inhibitory effects of tannins against 2nd instar larvae of *Spodoptera littoralis* were determined using a semi-artificial diet method described by El-Aswad et al. (2004). The isolated tannins solutions (in acetone) were incorporated with the artificial diet to prepare a range of concentrations (500, 250, 125 and 50 µg/g). Control diet was prepared with the maximum

amount of acetone alone used in the treated diet (0.5% v/w). After evaporation of acetone, 2 g of treated diet and 20 pre-weighed second-instar larvae were placed in each plastic box (20 cm length × 10 cm width × 10 cm depth). Three replicates were carried out at each concentration. The bioassay experiment terminated after the larvae of control had developed to pupae. The mortality percentages were recorded with time and the diet consumed by each larva was weekly determined by weighing the remaining diet of each treatment. The percentage antifeedant activity was calculated from the following equation:

$$\text{Antifeedant \%} = ((C - T)/C) * 100$$

where *C* is the weight of diet consumed in control and *T* is the weight of diet consumed in the treatment (El-Aswad et al. 2004).

Additionally, larval growth inhibition was weekly assayed relative to control based on larval weight gain of feeding on the treated diet. The growth inhibition was calculated from the following equation:

$$\text{Growth inhibition} = ((CL - TL)/CL) * 100$$

where CL is the larval weight gained in the control and TL is the larval weight gained in the treatment (El-Aswad et al. 2004).

Accordingly, EC₅₀ based on antifeedant activity and growth inhibition were calculated.

Statistical analysis

The toxicity index LC₅₀ and LT₅₀ were calculated and subjected to Probit analysis according to Finney (1952). EC₅₀ based on antifeedant activity and growth inhibition and RT₅₀ based on repellent action were also determined. Relative potency ratios at *P* = 0.05 were evaluated according to Robertson et al. (2017) using Polo plus program (LeOra 2003). The data of biological activity and total tannin content were subjected to analysis of variance (ANOVA). All the data of condensed and hydrolysable tannins presented as means ± SD were also subjected to analysis of variance (ANOVA) following a factorial split plot design with extract type allocated as main plots and tannin type as subplots. The differences between treatments were determined by Tukey's Honestly Significant Different test at *P* = 0.05 using the SAS, 9.4 software (SAS 2017).

Table 1 Extraction yield of crude tannins and total tannin content in different samples

Samples	Crude tannins extract/ dry sample (g/100 g)	Total tannins/ crude tannins extract (g/100 g)		Total tannins/ dry sample (g/100 g)	
		SD		SD	
Urtica	6.0	3.347 ^a	± 0.216	0.201 ^b	± 0.013
Bean hull	10.7	3.694 ^a	± 0.314	0.395 ^a	± 0.034
Black tea	15.7	2.417 ^b	± 0.236	0.379 ^a	± 0.037
Green tea	13.3	2.514 ^b	± 0.295	0.334 ^a	± 0.039

Total tannin contents within a column followed by the same letter are not significantly different ($p < 0.05$, based on Tukey, HSD test)

Results and discussion

Tannins study

Tannins characterization

The crude tannins content was extracted using Soxhlet apparatus from dry sample matters (urtica, bean hull, black tea and green tea). Consequently, the total tannins were determined in the crude extract. Table 1 illustrates that BT had the highest quantity of crude tannins extract (15.7 g/100 g dry matter) followed by GT (13.3 g/100 g dry matter). These results agree with those reported in the literature (Dalluge and Nelson 2000; Sakanaka et al. 1989). Total tannins were highest in bean hull based on crude extract or original dry sample (3.694 (± 0.314) & 0.395 (± 0.034) g/100 g). The results for total tannins in crude tannins extract were in order of B > U > GT > BT while that in dry matter were in order of B > BT > GT > U. In general, the quantity of crude tannins extract from black and green tea was higher than that extracted from urtica and bean, while the total tannins determined in crude tannins of black and green tea was lower than that of urtica and bean.

The condensed and hydrolysable tannins in this work was represented as absorbance units per mg of tannin fraction (Fig. 1). In each sample, the amount of hydrolysable tannins extracted was greater than the amount of condensed tannins. Green tea possessed the highest content of both condensed and hydrolysable fraction tannins (0.0123, 0.0215) determined using vanillin method (Price et al. 1978) and potassium iodate test (Bossu et al. 2006), respectively. Consequently, the statistical differences were recorded among all values of condensed and hydrolysable tannins. Several authors have reported by the existence of tannins in leguminous seeds (Ariga and Hamano 1990; Ariga et al. 1988), in Urtica plant (Jimoh et al. 2010; Joshi et al. 2014) and in black and green tea (Dalluge and Nelson 2000). The content of tannins in tannin fractions separated in this study from

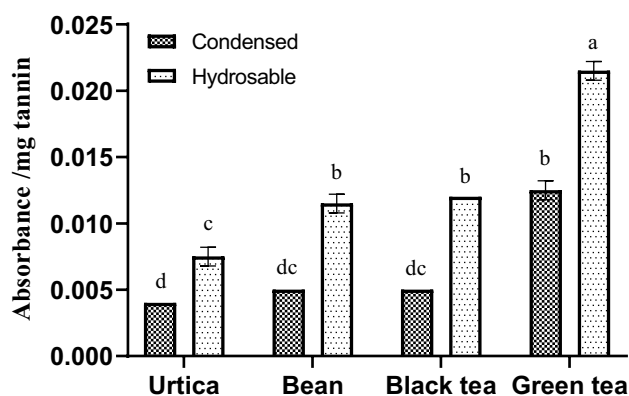


Fig. 1 Content of condensed and hydrolysable tannins expressed as absorbance units per mg tannin fraction. Means bars followed by the same letter at the top of error bars are not significantly different ($P \leq 0.05$) according to Tukey HSD test

beans was lower than that reported by Karamac et al. (2007) (0.195 absorbance units at 500 nm /mg).

Bioassay of tannins

Mortality effect

The mortality effects of isolated tannins from different sources on *Spodopetra littoralis* were evaluated within four weeks of feeding on treated diet and the lethal concentration required to kill 50% of the insect's population was calculated (Table 2). Initially (1st week), the effect of tannins from different sources was light, therefore the LC₅₀ of urtica weed could not be determined since at highest concentration (500 µg/g), the mortality effect did not exceed 50% of the insect's population. However, with time, the mortality effects of tannins from urtica weed gained traction and in the 4th week, 100% mortality was recorded at all concentrations, as was the case for green tea extracts too. Tannins extract from the other sources also showed a greater potency effect with time with beans extract having a LC₅₀ = 0.3 µg/g followed by black tea (LC₅₀ = 0.7 µg/g). This suggests that mortality effect of tannins might be due to other reasons rather than direct toxicity. Many researchers have previously illustrated the role of wild plant extracts in reducing damage of cotton leafworm (Abd El-Aziz and Ezz El-Din 2007).

Antifeedant activity

The quantity of diet consumed, the antifeedant percentages and EC₅₀ values based on the antifeedant percentages of the isolated tannins after three weeks of feeding on treated diet are shown in Table 3 and Fig. 2. The treatments reduced insect food consumption by altering insect's behavior, through direct action on the peripheral sensilla of insects

Table 2 LC₅₀ (µg/g) values of tannins isolated from different sources on *Spodoptera littoralis* within four weeks of feeding on treated diet

Time	U		B		BT		GT	
(weeks)	LC ₅₀ (Fiducial limits)	LC ₅₀ ratio test	LC ₅₀ (Fiducial limits)	LC ₅₀ ratio test	LC ₅₀ (Fiducial limits)	LC ₅₀ ratio test	LC ₅₀ (Fiducial limits)	LC ₅₀ ratio test
1	–a	–	146.1 (98.482– 270.755)	B vs BT* B vs GT ^{ns}	343.2 (230.766– 718.119)	BT vs B* BT vs GT*	105.5 (89.97– 123.71)	GT vs B ^{ns} GT vs BT*
2	93.0 (21.166– 163.826)	U vs B* U vs BT ^{ns} U vs GT*	2.6 (1.553– 4.387)	B vs U* B vs BT* B vs GT*	55.7 (4.329– 106.719)	BT vs U ^{ns} BT vs B* BT vs GT ^{ns}	29.2 (9.939– 44.29)	GT vs U* GT vs B* GT vs BT ^{ns}
3	10.8 (0.548– 28.376)	U vs B ^{ns} U vs BT ^{ns} U vs GT ^{ns}	3.5 (1.730– 6.956)	B vs U ^{ns} B vs BT ^{ns} B vs GT ^{ns}	11.5 (0.245– 30.672)	BT vs U ^{ns} BT vs B ^{ns} BT vs GT ^{ns}	17.1 (3.975– 30.286)	GT vs U ^{ns} GT vs B ^{ns} GT vs BT ^{ns}
4	–b	–	0.3 (0.07–1.379)	B vs BT ^{ns}	0.7 (0.1001– 5.137)	BT vs B ^{ns}	–b	–

^aNot calculated because the highest mortality percentage is not reach to 50%

^bNot calculated because the mortality percentages for all concentrations are 100%

Asterisk indicates significant difference ($p < 0.05$) whereas "ns" means not significant ($p > 0.05$) between LC_{50s} per each week

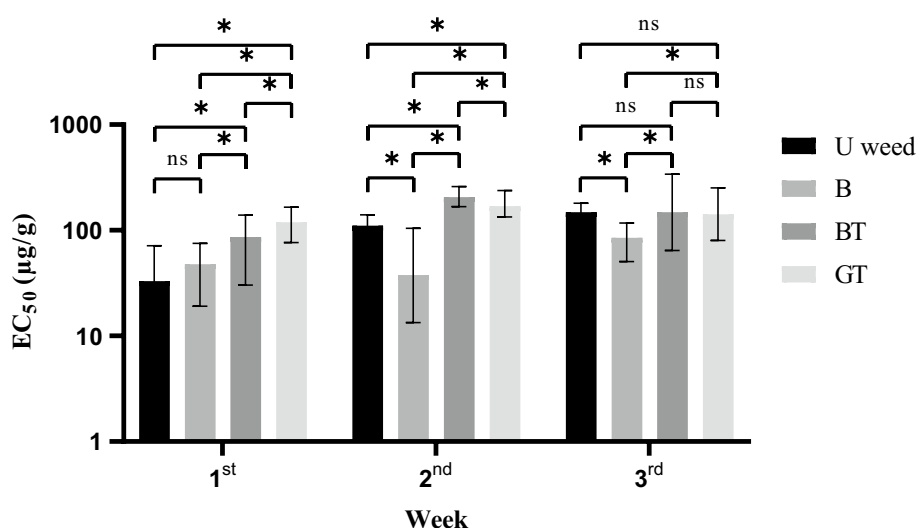
Table 3 Antifeedant activity of tannins isolated from different sources on *Spodoptera littoralis* within three weeks of feeding on treated diet

Time (weeks)	Conc. (µg/g)	U		B		BT		GT	
		Diet con- sumed mg/ larva (± SD)	Antifeedant (%)	Diet con- sumed mg/ larva (± SD)	Antifeedant (%)	Diet con- sumed mg/ larva (± SD)	Antifeedant (%)	Diet con- sumed mg/ larva (± SD)	Antifeedant (%)
1 st	500	76.8 (± 2.12)	72.2 a	60.0 (± 2.83)	78.2 a	88.4 (± 4.29)	68.0 a	65.0 (± 1.41)	76.5 a
	250	84.1 (± 7.21)	69.5 a	72.1 (± 1.69)	73.9 a	110.1 (± 4.10)	60.1 a	124.9 (± 1.24)	54.8 b
	125	107.5 (± 10.60)	61.0 ab	79.9 (± 5.75)	71.1 a	123.6 (± 10.47)	55.2 ab	146.0 (± 1.41)	47.2 c
	50	127.9 (± 1.27)	53.7 b	148.5 (± 4.95)	46.2 b	155.5 (± 9.19)	43.7 b	163.0 (± 4.24)	41.0 d
	0	276.5 (± 10.61)	0.0 c	276.5 (± 10.61)	0.0 c	276.5 (± 10.61)	0.0 c	276.5 (± 10.61)	0.0 e
2 nd	500	54.5 (± 3.54)	79.2 a	– ^b	– ^b	95.0 (± 7.07)	63.8 a	– ^b	– ^b
	250	75.5 (± 7.78)	71.2 a	86.1 (± 2.92)	67.2 a	132.5 (± 2.12)	49.4 b	120.0 (± 2.83)	54.2 a
	125	141.5 (± 3.54)	46.0 b	91.1 (± 1.24)	65.3 a	145.5 (± 3.54)	44.5 bc	142.5 (± 3.54)	45.6 b
	50	167.4 (± 11.86)	36.1 b	126.7 (± 3.86)	51.6 b	154.4 (± 1.04)	41.1 c	153.7 (± 6.61)	41.4 b
	0	262.0 (± 4.24)	0.0 c	262.0 (± 4.24)	0.0 c	262.0 (± 4.24)	0.0 d	262.0 (± 4.24)	0.0 c
3 rd	500	59.5 (± 0.71)	76.2 a	– ^b	– ^b	101.5 (± 2.12)	59.4 a	– ^b	– ^b
	250	72.5 (± 10.61)	71.0 a	86.3 (± 3.3)	65.5 a	122.5 (± 4.95)	51.0 b	105.0 (± 7.07)	58.0 a
	125	153.5 (± 2.12)	38.6 b	94.5 (± 0.71)	62.2 b	132.0 (± 2.83)	47.2 bc	137.5 (± 3.54)	45.0 b
	50	185.4 (± 7.66)	25.8 c	153.9 (± 5.54)	38.4 c	137.1 (± 1.44)	45.1 c	147.8 (± 4.10)	40.9 b
	0	250.0 (± 4.95)	0.0 d	250.0 (± 4.95)	0.0 d	250.0 (± 4.95)	0.0 d	250.0 (± 4.95)	0.0 c

^aAntifeedant percentages for each week within a column followed by the same letter are not significantly different ($p < 0.05$, based on the LSD test)

^bNot tested

Fig. 2 Antifeedant activity of urtica weed (U), bean hull (B), black tea (BT) and green tea (GT) expressed as EC_{50} ($\mu\text{g/g}$) with bars representing upper and lower limits against *Spodoptera littoralis* within three weeks. Asterisk indicates significant difference ($p < 0.05$) whereas "ns" means not significant ($p < 0.05$) between treatments per each week



(Isman 2002). All the tannins extract showed strong antifeedant activity at the higher concentration with a narrow standard deviation margin and a weak antifeedant activity at lower concentrations. Marginal significant differences were observed at various concentrations of the same treatment (mainly at lower concentration) with a greater significant difference observed at different concentrations of tannins extracted from GT. In other study, it was concluded that on addition of artificial diet, the manner of limonoids antifeedant activity was dependent on its concentration (El-Aswad et al. 2004). During the first week of treatment, tannins extracted from U presented the strongest antifeedant activity with an EC_{50} of 33.034 $\mu\text{g/g}$ followed by tannins extracted from beans with an EC_{50} of 47.839 $\mu\text{g/g}$. In the second and third week, tannins isolated from B showed the highest antifeedant activity (EC_{50} = 37.733 and 84.828, respectively) compared to tannins extracted from U (EC_{50} = 110.288 and 147.922, respectively). Treatments of BT and GT however, continued to show weaker antifeedant activity with the former being weakest in the subsequent weeks. We could not however evaluate the amount of diet consumed and consequently the antifeedant percentages of compounds B and GT in the 2nd and 3rd week due to the death of the insects, this coincides with the findings of others such as Ariga and Hamano (1990) and Dalluge and Nelson (2000).

Tannins at high concentration may act as antinutritional. They are astringent and efficient feeding deterrents to plenty of insects by binding to the proteins, reducing the efficacy to absorb nutrients and by causing midgut lesions (Dubey et al. 2016; Khasnabis et al. 2015). However, it is concluded that while tannins do act as antifeedants, there is little evidence supporting the theory that the underlying cause of this is digestibility reduction (Mole and Waterman 1987). Ethanol extract of *Vernonia ocephala* containing many phytochemicals such as tannins was most potent as feeding inhibition

against *Tribolium castaneum*. These findings illustrate the possible use of extract for developing effective botanical pesticides in pest management (Aliyu et al. 2014). Furthermore, many authors discovered that the antifeedant effect for larvae of cotton leafworm may also be due to the chemical composition of plants such as alkaloids, flavonoids, terpenes and tannins (Abdallah et al. 2017; Abdel-Rahman and Al-Mozini 2007; Adeyemi and Mohammed 2014; Elsharkawy et al. 2018).

Growth inhibition activity

Table 4 and Fig. 3 elucidates the growth inhibition activities of the isolated tannins on *S. littoralis* larvae. Generally, during the 3 weeks of feeding, larval growth inhibition was greater at higher concentrations and weaker at lower concentrations. During the first week of treatment, tannins extract from U demonstrated the strongest growth inhibition with an EC_{50} of 24.824 $\mu\text{g/g}$ followed by tannins extracted from BT, GT, B having EC_{50} of 37.415, 144.895, 197.823 $\mu\text{g/g}$, respectively. However, in the second and third week, the growth inhibition ability of tannins isolated from U slightly decreased, with an EC_{50} of 55.549 $\mu\text{g/g}$ and 34.596 $\mu\text{g/g}$, discretely, while the growth inhibition ability of tannins from other treatments slightly increased with time as compared to the first week. The results for growth inhibition at 3rd week were in order of B (EC_{50} = 2.23) > BT (LC_{50} = 13.042) > U (LC_{50} = 34.596) > GT (EC_{50} = 45.132). Many studies have demonstrated that dietary tannin can reduce growth and fecundity of some insect species (Awmack and Leather 2002; Bala et al. 2018; Ma et al. 2019; Mueller-Harvey et al. 2019). However, one of the defense strategies of the plant leaves against the aphid *Melaphis chinensis* (Aphididae) attack involves the rapid accumulation of tannic

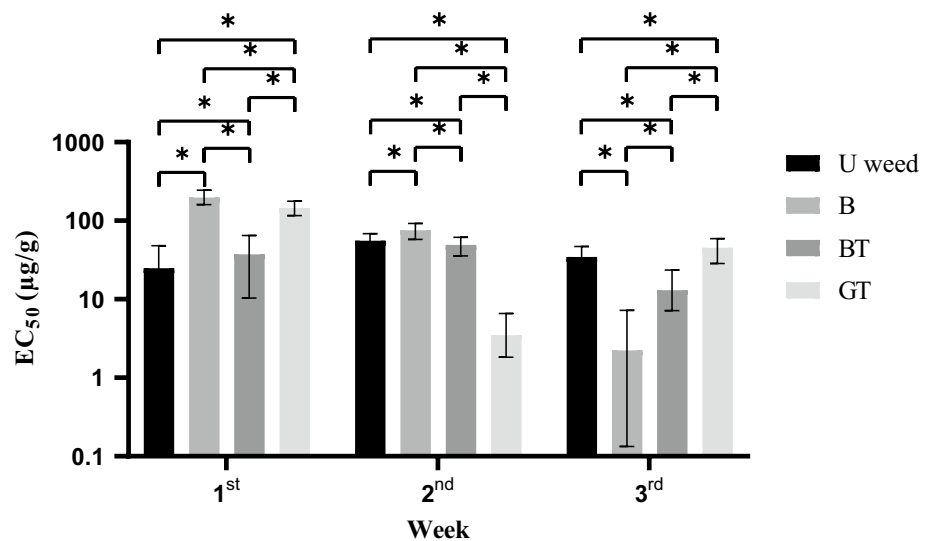
Table 4 Growth inhibitory action of tannins isolated from different sources on *Spodoptera littoralis* within three weeks of feeding on treated diet

Time (week)	Conc. ($\mu\text{g/g}$)	U		B		BT		GT	
		Mean weight gain mg/larva ($\pm$ SD)	Growth inhibition (%)	Mean weight gain mg/larva ($\pm$ SD)	Growth inhibition (%)	Mean weight gain mg/larva ($\pm$ SD)	Growth inhibition (%)	Mean weight gain mg/larva ($\pm$ SD)	Growth inhibition (%)
1 st	500	2.4 ($\pm$ 0.35)	82.6 a	1.8 ($\pm$ 0.28)	87.3 a	3.0 ($\pm$ 0.14)	78.6 a	3.2 ($\pm$ 0.71)	76.6 a
	250	2.5 ($\pm$ 0.70)	81.6 a	8.7 ($\pm$ 1.0)	36.9 b	3.3 ($\pm$ 0.96)	75.7 a	5.3 ($\pm$ 0.27)	62.0 a
	125	4.0 ($\pm$ 0.95)	70.7 a	9.7 ($\pm$ 0.82)	31.1 b	4.3 ($\pm$ 0.02)	69.4 a	6.4 ($\pm$ 0.41)	54.4 a
	50	5.8 ($\pm$ 0.20)	58.5 a	9.9 ($\pm$ 0.32)	29.2 b	6.8 ($\pm$ 0.01)	51.2 a	11.2 ($\pm$ 1.51)	20.7 b
	0	14.2 ($\pm$ 2.75)	0.0 b	14.2 ($\pm$ 2.75)	0.0 b	14.2 ($\pm$ 2.75)	0.0 b	14.2 ($\pm$ 2.75)	0.0 b
2 nd	500	1.1 ($\pm$ 0.07)	97.2 a	— ^b	— ^b	1.1 ($\pm$ 0.71)	97.1 a	— ^b	— ^b
	250	3.0 ($\pm$ 0.09)	91.8 a	14.5 ($\pm$ 1.00)	60.6 a	2.5 ($\pm$ 0.41)	93.3 a	1.5 ($\pm$ 0.72)	95.8 a
	125	3.5 ($\pm$ 0.71)	90.6 a	16.2 ($\pm$ 1.87)	56.0 b	7.0 ($\pm$ 0.57)	81.0 b	5.2 ($\pm$ 0.64)	85.9 b
	50	22.1 ($\pm$ 2.15)	39.5 b	20.1 ($\pm$ 1.66)	45.5 c	18.6 ($\pm$ 0.54)	49.4 c	10.5 ($\pm$ 0.65)	71.4 c
	0	36.9 ($\pm$ 3.82)	0.0 c	36.9 ($\pm$ 3.82)	0.0 d	36.9 ($\pm$ 3.82)	0.0 d	36.9 ($\pm$ 3.82)	0.0 d
3 rd	500	1.0 ($\pm$ 0.28)	97.4 a	— ^b	— ^b	1.03 ($\pm$ 0.39)	97.3 a	— ^b	— ^b
	250	1.5 ($\pm$ 0.68)	96.1 a	7.5 ($\pm$ 0.28)	80.7 a	2.0 ($\pm$ 0.14)	94.9 a	3.5 ($\pm$ 0.71)	91.0 a
	125	4.5 ($\pm$ 0.28)	88.5 b	9.6 ($\pm$ 0.71)	75.4 a	3.5 ($\pm$ 0.81)	90.9 a	8.9 ($\pm$ 0.21)	77.2 a
	50	17.3 ($\pm$ 0.35)	55.71 c	11.0 ($\pm$ 0.99)	71.6 a	12.0 ($\pm$ 0.31)	69.2 b	18.00 ($\pm$ 1.43)	53.6 b
	0	39.02 ($\pm$ 2.81)	0.0 d	39.02 ($\pm$ 2.81)	0.0 b	39.02 ($\pm$ 2.81)	0.0 c	39.02 ($\pm$ 2.81)	0.0 c

^aGrowth inhibition percentages for each week within a column followed by the same letter are not significantly different ($p < 0.05$, based on the LSD test)

^bNot tested

Fig. 3 Growth inhibitory action ($\text{EC}_{50} \pm$ upper and lower limits; $\mu\text{g/g}$) of tannins isolated from urtica weed (U), bean hull (B), black tea (BT) and green tea (GT) on *Spodoptera littoralis* within three weeks. Asterisk indicates significant difference ($p < 0.05$) between treatments per each week



acid which forms galls along the midrib of the leaves. In response, the aphid would detoxify the ingested toxic tannic acid to relatively nontoxic garlic acid, whereas cotton pink bollworm *Pectinophora gossypiella* larvae remain sensitive to the ingested tannic acid (Kubo et al. 2003). Also, it was reported that the growth of *Spodoptera litura* was inhibited by tannin and that the inhibitory effect was proportional to the quantity of tannin ingested. These results confirm the traditional theory that plant tannin is

a protective agent against generalist herbivores, and that its influences are dependent on its concentration in foliage (Nomura and Itioka 2002).

Onion essential oil study

Onion essential oil characterization

While analyzing the composition of the volatile essential oil by gas chromatography/mass spectrometry, 30 chemicals constituting 72.78% of the entire oil composition was found. The volatile components of essential oils can be grouped into terpenes, benzene derivatives, hydrocarbons and other miscellaneous compounds (Guenther and Althausen 1948; Ngoh et al. 1998; Tripathi et al. 2009). The volatile nature of compounds in onion was studied by many researchers

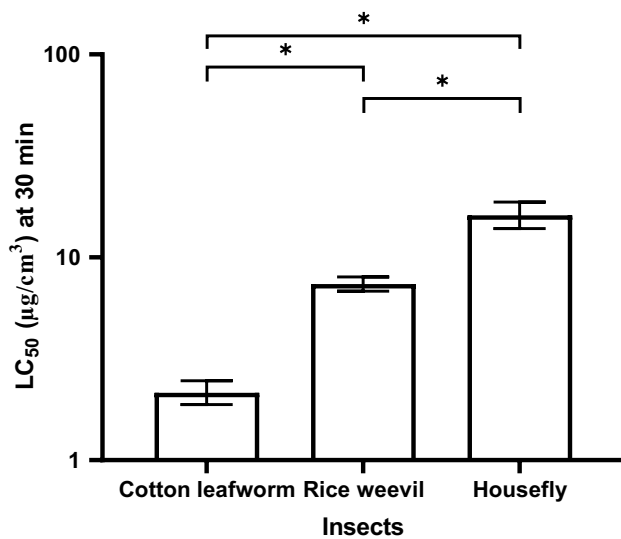
(Brodnitz et al. 1969; Lukes 1971; Mazza et al. 1980). Furthermore, other studies have also demonstrated the presence of sulfur in onion compounds (Blank 2002; El-Wakil et al. 2015; Ueda et al. 1994) which coincides with our studies where we detected 12 organosulfur compounds. According to our results in Table 5, ten of these chemical compounds had one or more peak area percentage, particularly, mercaptamine (23.27%), amino methane sulfonic acid (15.00%), 1-(methyl thio) but-1-yn-4-ol (6.49%), 1,3,4-Thiadiazolidine 2,5-dithione (3.38%), 2-Bromoethyl vinyl sulfide (3.31%). Other studies of the chemical composition of *Allium cepa* are consistent with the results of our study though slight

Table 5 Chemical composition of onion essential oil expressed as relatively percentage

Rt	Chemical compound	Area %	Molecular Formula	MW
2.76	21H,23H-Porphine, 2,7,12,17-tetraethyl-22,24-dihydro-3,8,13,18,21,23-hexamethyl-	1.01	C ₃₄ H ₄₄ N ₄	508
3.36	Acetic acid, 17-(4-chloro-5-methoxy-1,5-dimethylhexyl)-4,4,10,13,14-pentamethyl-2,3,4,5,6,7,10,11,12,13,14,15,16,17-tetradecahydro-1-phenanthryl-	0.83	C ₃₃ H ₅₅ ClO ₃	534
4.10	2-Bromoethyl vinyl sulfide	3.31	C ₄ H ₇ BrS	166
4.53	L-Valine, N-[N,O-bis(2,4-dinitrophenyl)-L-tyrosyl]-,methyl ester	0.49	C ₂₇ H ₂₆ N ₆ O ₁₂	626
5.25	Styrene	0.54	C ₈ H ₈	104
5.76	3-(7,8,12,13,17,18-Hexaethyl-3-methyl-22, 24-dihydro-porphin-2-yl)-propionitrile	0.77	C ₃₆ H ₄₃ N ₅	545
6.90	Difuro[2',3':5,6:3'',2'':7,8]perlylo[1,12-def][1,3]dioxepin-8,15-dione, 10,11,12,13-tetrahydro-1,7-dihydroxy-10,13-dimethyl-	0.42	C ₂₇ H ₁₈ O ₈	470
8.28	1,3,4-Thiadiazolidine2,5-dithione	3.38	C ₂ H ₂ N ₂ S ₃	150
9.32	Aminomethanesulfonic acid	15.00	CH ₅ NO ₃ S	111
11.34	3-Methylthio-6-methyl-5-thioxo-2,5-dihydro-1,2,4-triazine	1.37	C ₅ H ₇ N ₃ S ₂	173
11.43	2-Hydroxyethyl vinyl sulfide	0.30	C ₄ H ₈ OS	104
11.49	Thiirane	0.79	C ₂ H ₄ S	60
13.89	1-(Methylthio)but-1-yn-4-ol	6.49	C ₅ H ₈ OS	116
14.20	Mercaptamine	23.27	C ₂ H ₇ NS	77
17.41	Sumatriptan	0.65	C ₁₄ H ₂₁ N ₃ O ₂ S	295
23.47	4-Amino-3-mercapto-1,2,4-triazole	0.31	C ₂ H ₄ N ₄ S	116
25.07	Trifluoromethylperoxynitrate	0.91	CF ₃ NO ₄	147
27.02	Suberic acid	0.55	C ₈ H ₁₄ O ₄	174
28.84	2-Chloroethyl methylsulfoxide	1.21	C ₃ H ₇ ClOS	126
29.43	Hexadecanamide, N,N-bis{[2-(2-butoxyethoxy)ethoxy]carbonylmethyl}-	0.33	C ₃₆ H ₆₉ NO ₉	659
34.59	2,2-Bis[4-[(4,6-dichloro-1,3,5-triazin-2-yl)oxy]phenyl]propane	0.60	C ₂₁ H ₁₄ Cl ₄ N ₆ O ₂	522
35.94	2-Propenenitrile	0.70	C ₃ H ₃ N	53
36.34	1,4-Ethenoanthracene-2,3-dicarboxylicacid, 1,4-dihydro-9,10-diphenyl-, dimethyl ester	0.29	C ₃₂ H ₂₄ O ₄	472
36.98	Lycopene	0.79	C ₄₀ H ₅₆	536
37.44	Lycoxanthin	1.00	C ₄₀ H ₅₆ O	552
38.31	Hematoporphyrin ix	0.44	C ₃₄ H ₃₈ N ₄ O ₆	598
42.42	L-Proline, 1-[O-(1-oxohexyl)-N-[N-[N6-(1-oxohexyl)-N2-[N-(1-oxohexyl)-L-valyl]-L-lysyl]-L-valyl]-L-tyrosyl]-,methyl ester	0.52	C ₄₉ H ₈₀ N ₆ O ₁₀	912
42.83	Pyridine, 3-(butylthio)-	0.96	C ₉ H ₁₃ NS	167
43.35	Hexadecanoic acid, 1a,2,5,5a,6,9,10,10a-octahydro-5a-hydroxy-4-(hydroxymethyl)-1,1,7,9-tetramethyl-6,11dioxo-1H-2,8a-methanocyclopenta[a] cyclo propa[e] cyclodecen-5-yl ester,[1aR-(1aà,2a,5a,5aa,8aà,9a,10aà)]-	1.00	C ₃₆ H ₅₆ O ₆	584
46.52	Methadone	0.60	C ₂₁ H ₂₇ NO	309
Total		72.78		
Minor compounds		27.22		

Table 6 Mortality percentages of some insects exposed to different concentrations of onion essential oil and their toxicity index

Conc. (µg/cm³)	Cotton leafworm									Rice weevil						Housefly					
	Exposure time (min)																				
	10	30	60	10	30	60	90	120	180	10	30	60	90	120	180	10	30	60	90	120	180
	Mortality (%)																				
1	0	25	100	0	0	10	10	10	20	0	0	0	0	20	30						
2.5	0	50	100	0	10	20	40	50	70	0	5	50	100								
5	25	75	100	0	20	30	60	90	100	0	25	70	100								
7.5	50	95	100	10	40	40	80	100		0	35	80	100								
10	75	100		60	75	80	100			0	40	85	100								
15	100			80	90	100				10	45	90	100								
25				100	100					15	50	100									
50										50	85	100									

**Fig. 4** Fumigant toxicity of onion essential oil on cotton leafworm, rice weevil and housefly insects expressed as LC_{50} ($\mu\text{g}/\text{cm}^3$) with bars representing upper and lower limits. Asterisk indicates significant difference ($p < 0.05$) between insects

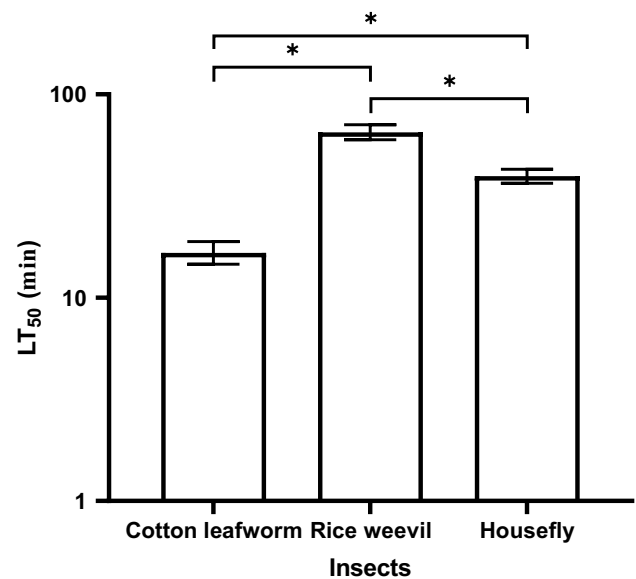
differences were observed in components percentages (Hosoda et al. 2003; Vazquez-Armenta et al. 2016). This could be attributed to the differences in either geographical or environmental factors (Perry et al. 1999; Yassen and Khalid 2009).

Bioassay of onion essential oil

Mortality effect

Mortality percentage of onion essential oil on different insects at various concentrations and time was evaluated using fumigation technique as observed in Table 6.

Overall, mortality percentage increased with both an increase in concentration and exposure time. The mortality

**Fig. 5** LT_{50} (min) values of onion essential oil ($5 \mu\text{g}/\text{cm}^3$) on cotton leafworm, rice weevil and housefly insects with bars representing upper and lower limits. Asterisk indicates significant difference ($p < 0.05$) between insects

in control was less than 5%. According to Fig. 4, the LC_{50} ($\mu\text{g}/\text{cm}^3$) at 30 min reflected that the onion essential oil was more toxic to cotton leafworm with a $\text{LC}_{50} = 2.15 \mu\text{g}/\text{cm}^3$ compared to rice weevil ($\text{LC}_{50} = 7.38$) and house flies ($\text{LC}_{50} = 16.09$).

As shown in Fig. 5, the LT_{50} at concentration of $5 \mu\text{g}/\text{cm}^3$ showed that onion oil was more potent in cotton leafworm compared to other insects. The mortality percentage reached 100% for cotton leafworm, housefly and rice weevil after 60, 90, and 180 min of exposure time, respectively. This implies that it takes a shorter time for onion essential oil to kill cotton leafworm compared to house flies, rice weevil, consecutively. EOs are hydrophobic in nature and can penetrate the

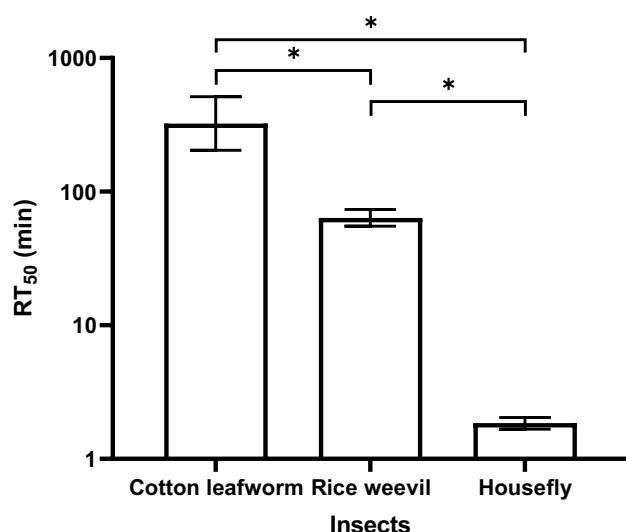


Fig. 6 Repellency action of onion essential oil ($10 \mu\text{g}/\text{cm}^3$) expressed as RT_{50} (min) $\pm$ upper and lower confidence limits against tested insects. Asterisks represent statistical significance ($P < 0.05$)

insect resulting in biochemical disorder causing mortality. The route of entry and mechanism of its action are some the factors that influences toxicity (Ozols and Bicevskis 1979). Clearly, it was demonstrated that essential oils could be applicable to the management of insect pests (Ebadollahi et al. 2013). In general, a few studies including Oparaek et al. (1999) and Stoll (1988) have reported potency of *Allium* essential oil against insects. However, insufficient studies on the bioactivity of essential oil from *Allium* species against insects, especially their larvae.

Repellency action of the essential oil

The RT_{50} (repellent time required to deter away 50% of insect population from the treated space in WHO tube using a concentration of $10 \mu\text{g}/\text{cm}^3$) was seen to be highly effective in houseflies ($\text{RT}_{50} = 1.85$ min), followed by rice weevil ($\text{RT}_{50} = 63.67$ min) and cotton leafworm ($\text{RT}_{50} = 322.63$ min) with a significant difference among tested insects (Fig. 6). This could be due to ease in movement (escape response) of the houseflies as compared to the other insects. This result agrees with the results of other study which indicated that some species of the genus *Allium* have consequently shown repellent and insecticidal properties (Hosoda et al. 2003; Vazquez-Armenta et al. 2014, 2016).

Conclusion

The results of the experiments clearly demonstrated that isolated tannins from urtica weed, bean, black tea and green tea when added to diet at concentration from 50 to 500 $\mu\text{g}/\text{g}$

revealed strong antifeedant activity and growth inhibition action against *S. littoralis*. More studies on chemistry and modes of action of tannins could potentially provide novel insect control agents needed in pest management. These findings reflect the possibilities of using these compounds in insect control and further work on this is recommended. Moreover, onion essential oil has demonstrated toxicity efficacy and repellence action against a range of insect pests, field pest cotton leafworm, stored product pest rice weevil and domestic pest houseflies using fumigation technique. Therefore, this oil may be applied as fumigant or direct spray with a scope of effects from lethal toxicity to repellence in insects particularly in product storages and households. These features indicate that tannins as antifeedants and essential oil obtained from *Allium cepa* are becoming increasingly important as potential components of IPM strategies if the cost-effective problems will be solved. Green house and field studies would be required to determine if these bioactive chemicals tannins and onion essential oil are viable for insect control. Ultimately, the effects on end-use quality, lingering off-odors or taste and risk to humans would need to be determined before commercialization because, there is need for effective, renewable, nonpersistent in the environment and relatively safe to nontarget organisms and humans.

Funding Open access funding provided by The Science, Technology & Innovation Funding Authority (STDF) in cooperation with The Egyptian Knowledge Bank (EKB).

Declarations

Conflict of interest We wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

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