Potential Insecticidal Activity of Four Essential Oils against the Rice Weevil, *Sitophilus oryzae* (L.) (Coleoptera: Curculionidae)

Mohamed E. Tawfeek,[a] Hayssam M. Ali,[b] Mohammad Akrami,[c] and Mohamed Z. M. Salem [d,*]

Oils extracted from *Cymbopogon citratus*, *Lantana camara*, *Artemisia camphorata*, and *Imperata cylindrica* plants were used as potential insecticides against the rice weevil, *Sitophilus oryzae* (L.) (Coleoptera: Curculionidae). The phytochemical composition of the isolated oils was identified by gas chromatograph-mass spectrometry (GC-MS). Oil contact toxicities were evaluated against the adults of *S. oryzae*. The activities of acetylcholinesterase (AChE), alkaline phosphatase (ALP), and transaminases enzymes (AST) were measured. *L. camara* oil (LC_{50} = 9.81 mg/cm^2) demonstrated the highest effect, followed by *C. citratus* oil (LC_{50} = 10.89 mg/cm^2), *A. camphorata* EO (LC_{50} = 16.12 mg/cm^2), and *I. cylindrica* oil (LC_{50}= 36.85 mg/cm^2) against the adults of *S. oryzae*. The inhibition percentages of AChE were 38.8, 41.7, 35.0, and 27.2%; ALP were 42.4, 49.3, 28.1, and 18.7%; AST were 33.9, 38.7, 20.8, and 11.8%; and ALT were 22.7, 30.5, 14.6, and 9.6% after treated *S. oryzae* with oils from *C. citratus*, *L. camara*, *A. camphorata* and *I. cylindrica*, respectively. The highest abundant compounds in *C. citratus* were geranial (25.95%), nerylacetal (8.85%), and neral (8.45%), in *L. camara* were caryophyllene (12.2%), and 3-elemene (8.89%), in *A. camphorata* were germacrene D-4-ol (20.83%), and borneol (19.47%), and in *I. cylindrica* were 5-phenylundecane (10.68%), and 6-phenyldodecane (8.70%).

Keywords: Oily extracts; Chemical composition; Contact toxicity; Sitophilus oryzae; Acetylcholinesterase; Aspartate transaminase; Alanine transaminase

Contact information: a: Department of Applied Entomology and Zoology, Faculty of Agriculture, 21545 El-Shatby, Alexandria University, Alexandria, Egypt; b: Botany and Microbiology Department, College of Science, King Saud University, P.O. Box 2455, Riyadh 11451, Saudi Arabia c: Department of Engineering, University of Exeter, Exeter EX4 4QF, UK; d: Forestry and Wood Technology Department, Faculty of Agriculture (El-Shatby), Alexandria University, Alexandria 21545, Egypt;
** Corresponding Author: zidan_forest@yahoo.com*

INTRODUCTION

Stored-product pests cause critical misfortunes in weight and quality of the stored grains and cereal products (Fields and Korunic 2000; Neethirajan *et al.* 2007). The grain weevils (Curculionidae) are major pests of stored grains such as wheat, maize, and rice. The rice weevil, *Sitophilus oryzae* L. (Coleoptera: Curculionidae), is one of the most hazardous stored grain pests throughout the world (Pugazhvendan *et al.* 2009). Females deposit eggs into grain; larvae are legless and remain in the grain kernel for their entire duration. Feeding of *S. oryzae* larvae and adults can bring down grain weight by up to 75%, diminishing the dietary benefit and germination of the grains resulting in lower prices for seed grain (Dal Bello *et al.* 2000).

The protection of stored grain using synthetic pesticides is still the method of choice (Rattan 2010). However, many problems are associated with these chemicals, such as insect resistance, toxicity to mammals and other living organisms, toxic residues in stored products, increasing costs of application, and environmental contamination (Arthur *et al.* 2014). Therefore, there is interest in finding alternative ways for stored products protection. Natural pesticides with low mammalian toxicity are recommended for suppressing insect pests especially in storage (Parugrug and Roxas 2008).

Botanicals are plant-inferred materials that can be utilized as an important component in integrated pest management (IPM) for prevailing insect pests (Abdelsalam *et al.* 2019; Salem *et al.* 2020; Mosa *et al.* 2021; Salem *et al.* 2021; Moustafa *et al.* 2021). Botanical pesticides are biodegradable, have little or no deleterious impact on the environment and non-target living beings, cheap, easily produced, and may impede the advancement of resistance (Isman 2005; Rajendran and Sriranjini 2008). Essential oils (EOs) from different botanical parts have shown promising insecticidal activities. EOs from *Cymbopogon citratus*, *C. nardus*, and *Eucalyptus citriodora* have repellent effects against *Anopheles arabiensis* mosquitoes (Solomon *et al.* 2012). The EO composition of *C. citratus* EO showed the presence of neral, geranial, and *β*-pinene as main compounds, while in *C. nardus* they were citronellal, nerol, and citronellol; both oils showed low contact toxicity against *Dinoderus porcellus* L. (Coleoptera: Bostrichidae) (Loko *et al.* 2021). When tested against three stored grain insects, *Sitophilus oryzae, Rhyzopertha dominica*, and *Tribolium castaneum*, plant EOs could play an important role in control of stored-grain insects especially *S. oryzae* and could be recommended for use as a part of IPM program in stored grains (Tawfeek *et al.* 2017).

Acetylcholinesterase (AChE, EC 3.1.1.7) is a key enzyme that terminates nerve impulses by catalyzing the hydrolysis of neurotransmitter, acetylcholine, in the nervous system of various organisms. Its inhibition leads to paralysis and death (Zibaee 2011). Alkaline phosphatase (ALP, EC 3.1.3.1) is a set of hydrolytic enzymes, which hydrolyzes phosphomonoesters under alkaline conditions causing cytolysis of tissues during the insect development (Miao 2002). Aspartate aminotransferase (AST, EC 2.6.1.1) and alanine aminotransferase (ALT, EC 2.6.1.2) are also known as glutamate oxaloacetate transaminase (GOT) and glutamate pyruvate transaminase (GPT), respectively. The aminotransferases are important enzymes catalyzing amino acid catabolism and are critical in carbohydrate and protein metabolism (Zibaee *et al.* 2008). They are altered during various pathological and physiological activities (Etebari *et al.* 2005).

This work evaluated the insecticidal activities of the extracted oils from four plant species (*Cymbopogon citratus*, *Lantana camara*, *Artemisia camphorata*, and *Imperata cylindrica*) against *Sitophilus oryzae* (L.) adults. Their inhibitory effects on the enzyme activities of AChE, ALP, AST, and ALT were estimated.

EXPERIMENTAL

Insect Rearing

The rice weevil *Sitophilus oryzae* (L.) adults were gathered from infested wheat grains (*Triticum aestivum* L.). Insect rearing was performed at the laboratory of Applied Entomology and Zoology Department, Faculty of Agriculture (El-Shatby), Alexandria University, Alexandria, Egypt. The culture was maintained in plastic containers (26 × 30 × 20 cm) at 28 ± 2 °C and 65 ± 5 % relative humidity without exposure to any insecticide.

Extraction of Plant Oils

Fresh leaves collected from four plants and authenticated at the Department of Forestry and Wood Technology, Faculty of Agriculture, Alexandria University, Egypt, were used for the oil extraction. Oils from *Cymbopogon citratus*, *Lantana camara*, and *Artemisia camphorata* were extracted by the Clevenger method, where 100 g fresh weight from each material were cut to small pieces and put in 2-L flask containing 1000 mL of distilled water and subjected to hydrodistillation for 3 h to extract the essential oil (EO) (Salem *et al.* 2014, 2020). For *Imperata cylindrica*, 100 g were cut into small pieces, extracted by soaking in n-hexane solvent (200 mL) for 6 h under shaking, and filtered with Whatman No. 1 filter paper under suction pressure (Hamada *et al.* 2018). The *n*-hexane oily liquid extract (HOE) was separated and concentrated by evaporating the n-hexane solvent under vacuum using a rotary evaporator at 45 °C. The HOE was stored in brown tubes (Salem *et al.* 2019; Mohamed *et al.* 2020).

Contact Toxicity

The insecticidal activity of the four oils was tested against *S. oryzae* adults by the residual film technique (Qi and Burkholder 1981), under laboratory conditions at 12:12 h (light: dark photoperiod). Serial dilutions of tested oils from *C. citratus*, *L. camara*, *A. camphorata*, and *I. cylindrica* at 2, 5, 10, 20, 30, and 60 mg/cm^2 were prepared in acetone as a solvent. There were four replicates for each treatment in addition to controls. One mL of each concentration was placed on the bottom of each Petri dish (9 cm diameter). After the acetone was evaporated, 10 adult of rice weevils were placed into each dish. The same procedure was used for the control treated with acetone. Mortality percentages were recorded after 72 h of treatment. The LC_{50} and LC_{90} values as well as the slope of lines were calculated according to Finney (1952). It should be noted that, according to the residual film technique used in the bioassay test, the concentration is attributed to the area Unit. A unit of area means a spread rate at which one milligram of a substance is spread over the area of one square centimeter, which used as a dose calculation unit.

Biochemical Assays

Homogenate preparation

After treatment with LC_{50} values of the tested oils, *S. oryzae* adults were separately weighted and homogenized in 10 volumes (w/v) of ice cold 0.1 M phosphate buffer (pH 7.2) using a Teflon glass tissue homogenizer. In the ALP assay, the samples were homogenized in 10 volumes (w/v) of ice cold 0.1 M phosphate buffer (pH 9.8). The homogenates were centrifuged at 5000 rpm for 30 min at 4 °C. The obtained supernatants were divided into small portions and stored at -20 °C. Three replicates were used for each treatment.

Total soluble protein

The total protein content was determined calorimetrically according to the method of Lowry *et al*. (1951) by using bovine serum albumin (BSA) as a standard.

AChE activity

Acetylcholinesterase (AChE) activity was determined according to Ellman *et al.* (1961). The reaction mixture contained 2.78 mL of 0.1 M phosphate buffer (pH 8.0); 0.1 mL of ten times diluted DTNB reagent solution (39.5 mg of 5, 5-dithiobis-[2-nitro-benzoic acid] and 15 mg of sodium bicarbonate dissolved in 10 mL of 0.1 M phosphate buffer, pH

7.2); 0.1 mL of the supernatant; and 0.02 mL of 0.075 M acetylthiocholine iodide. The yellow color formed was measured after 10 min at 412 nm. Specific activity was expressed as µmol acetylthiocholine hydrolyzed/min/mg protein.

Alkaline phosphatase activity

Alkaline phosphatase (ALP) activity (Klin 1972) was determined using a diagnostic kit (Diamond Co., Cairo, Egypt). In this method, 20 µL of the enzyme source was added to 1000 µL of 1.0 M diethanolamine buffer (pH 9.8) containing 0.6 mM magnesium ions and 1 mM p-nitrophenyl phosphate, mixed in the cuvette, incubated for 30 sec. With the stopwatch started simultaneously, the output was read again after exactly 1, 2, and 3 min at 405 nm using a spectrophotometer (Sequoia-Turner Model 340; Texas City, TX, USA). Specific activity was expressed as IU/mg protein/hr.

Transaminases activity

Aspartate aminotransferase (AST) and alanine aminotransferase (ALT) activities were determined as previously described (Reitman and Frankel 1957). In this method, 100 µL of enzyme source was added to 500 µL of 0.1 M phosphate buffer (pH 7.2) containing 80 mM L-aspartate as a substrate for AST or 80 mM D-L-alanine as a substrate for ALT, and 4 mM ά-ketoglutarate.

After incubating the mixture for 30 min at 37 °C, 500 µL of developing color reagent (4 mM 2, 4-dinitrophenylhydrazine) was added, and the solution was incubated for 20 min at room temperature. Lastly, 5 mL of 0.4 N NaOH was added, and the mixture was left at room temperature for 5 min. An assay mixture without enzyme source was used as the blank, and the absorption was measured at 546 nm. AST and ALT specific activities were expressed as IU/mg protein/hr.

GC–MS Analysis of the Oils

The chemical composition of the extracted oils was analyzed with a Trace GC Ultraho-ISQ mass spectrometer (Thermo Scientific, Waltham, MA, USA) with a direct capillary column TG–5MS (30 m × 0.25 mm × 0.25 µm film thickness). Oils were diluted in *n*-hexane solvent (3 *n*-hexane: 1 oil) before being injected to the GC–MS. The used carrier gas was He (flow rate of 1 mL/min).

The program and oven temperatures conditions can be found in previous work (Moustafa *et al.* 2021). The Xcalibur 3.0 data system (Thermo Fisher Scientific) with the Match factor from the GC–MS literature is a very intelligent tool to identify chemical constituents, where the value ≥650 is acceptable to confirm the compounds (El-Sabrout *et al.* 2019; Behiry *et al.* 2020; Salem *et al.* 2020; Abd-Elkader *et al.* 2021; Ali *et al.* 2021; Moustafa *et al.* 2021).

Statistical Analysis

Means were compared for significance using one-way analysis of variance (ANOVA) test with Least Significant Difference ($LSD_{0.05}$) (2005). Mortality rates were corrected according to Abbott’s formula (Abbott 1925) and plotted against concentrations as log/probit regression lines. LC50, LC90 values, and the toxicity index, as well as the slope of the lines were calculated according to Finney (1952).

RESULTS AND DISCUSSION

Insecticidal Activity of the Extracted Oils

The effects of four natural plant oils from *Cymbopogon citratus*, *Lantana camara*, *Artemisia camphorata*, and *Imperata cylindrica* on the rice weevil *Sitophilus oryzae* adults were tested after 72 h from treatment by residual film technique. As shown in Table 1, among the four natural plant oils used, the *L. camara* EO exhibited the highest effect against the adults of *S. oryzae* (LC_{50} = 9.81 mg/cm^2), followed by *C. citratus* EO (LC_{50} = 10.89 mg/cm^2), *A. camphorata* EO (LC_{50} = 16.12 mg/cm^2), and *I. cylindrica* HOE (LC_{50}= 36.85 mg/cm^2).

Table 1. Contact Toxicity of the Oils against *S. oryzae* Adults after 72 h from Treatment by Residual Film Technique

Oil source	LC_{50}[a] (mg/cm^2)	95% CL[b] (mg/cm^2)	LC_{90}[c] (mg/cm^2)	95% CL (mg/cm^2)	Slope ± SE[d]	(χ^2)[e]	R^2
Cymbopogon citratus	10.89	7.23 to 16.38	40.77	27.08 to 61.37	2.27 ± 0.09	0.96	0.97
Lantana camara	9.81	6.55 to 14.69	41.20	27.52 to 61.68	2.32 ± 0.09	0.76	0.81
Artemisia camphorata	16.12	9.70 to 26.80	108.04	64.99 to 179.63	1.76 ± 0.11	0.53	0.69
Imperata cylindrical	36.85	20.49 to 66.29	223.99	124.52 to 402.94	1.70 ± 0.13	0.78	0.81

[a] The concentration causing 50% mortality. [b] The concentration causing 90% mortality. [c] Confidence limits [d] Slope of the concentration-mortality regression line ± standard error. [c] Chi square value.

In vivo Effect of Oils on AChE and ALP Activities in *S. oryzae* Adults

The changes in AChE and ALP enzymes activities in *S. oryzae* are displayed in Table 2. There was no significant difference in the AChE activity of *S. oryzae* adults after being treated with LC_{50} of four EOs. The inhibition percentages were 38.8, 41.7, 35.0, and 27.2% after treatment with Eos from *C. citratus*, *L. camara*, *A. camphorata*, and *I. cylindrica*, respectively, relative to the control. The inhibition percentages in ALP activity were 42.4, 49.3, 28.1, and 18.7% after treatment with *C. citratus*, *L. camara*, *A. camphorata*, and *I. cylindrica* oils, respectively, relative to the control.

Table 2. Changes in Acetylcholinesterase (AChE) and Alkaline Phosphatase (ALP) Activities of *S. oryzae* Adults as Affected by LC_{50} of Extracted Oils

Oil source	Conc. (mg/cm^2)	AChE activity (μmol/ min/mg protein) ± SD[1]	Change[2] (%)	ALP activity (IU/ mg protein/hr) ± SD	Change (%)
Control	-	32.0^a ± 2.6	-	945.7^a ± 8.4	-
Cymbopogon citratus	10.89	19.6^b ± 1.5	- 38.8	544.6^d ± 7.2	- 42.4
Lantana camara	9.81	18.7^b ± 0.5	- 41.7	479.3^e ± 11.5	- 49.3
Artemisia camphorata	16.12	20.8^b ± 0.5	- 35.0	680.4^c ± 10.8	- 28.1
Imperata cylindrica	36.85	23.3^b ± 1.2	- 27.2	768.5^b ± 6.3	- 18.7
$LSD_{0.05}$		4.66	-	16.54	-

Means followed by the same letters in the same column are not significantly different at $P \leq 0.05$.
[1]SD means standard deviation. [2] Change (%) = (treated – control) ÷ control × 100.

In vivo Effect of Oils on AST and ALT Activities in *S. oryzae* Adults

Table 3 shows the inhibitory effects in the activities of both AST and ALT in *S. oryzae* adults. The inhibition percentages of AST activity were 33.9, 38.7, 20.8, and 11.8% after being treated *S. oryzae* adults with *C. citratus*, *L. camara*, *A. camphorata*, and *I. cylindrica* oils, respectively, compared with the control. Meanwhile, the inhibition percentages of ALT activity were 22.7, 30.5, 14.6, and 9.6% after treating *S. oryzae* adults with the same previous extracts compared with the control.

Table 3. Changes in Aspartate Aminotransferase (AST) and Alanine Aminotransferase (ALT) Activities of *S. oryzae* Adults as Affected by LC_{50} of the Extracted Oils

Oil Source	Conc. (mg/cm^2)	AST activity (IU/ mg protein/hr) ± SD[1]	Change[2] (%)	ALT activity (IU/ mg protein/hr) ± SD	Change (%)
Control	-	542.2^a ± 6.9	-	753.0^a ± 3.4	-
Cymbopogon citratus	10.89	358.3^d ± 5.5	- 33.9	582.3^d ± 3.8	- 22.7
Lantana camara	9.81	332.4^e ± 6.2	- 38.7	523.2^e ± 4.2	- 30.5
Artemisia camphorata	16.12	429.3^c ± 6.5	- 20.8	642.8^c ± 5.6	- 14.6
Imperata cylindrica	36.85	478.5^b ± 5.8	- 11.8	680.6^b ± 5.6	- 9.6
$LSD_{0.05}$		11.35	-	8.44	-

Means followed by the same letters in the same column are not significantly different at $P \leq 0.05$.
[1]SD means standard deviation. [2] Change (%) = (treated – control) ÷ control × 100.

Chemical Composition of the Oils

Table 4 presents the chemical components of the EO from *C. citratus*, where the highest abundant compounds were geranial (25.95%), nerylacetal (8.85%), neral (8.45%), linalool oxide (6.36%), 2-furanmethanol (5.4%), *cis*-linalool oxide (4.29%), 10-hydroxygeraniol (3.92%), *cis*-verbenol (3.85%), *cis*-linalool oxide (2.38%), 4-methyl-valeric acid (2.25%), and sobrerol 8-acetate (2.19%).

Table 5 shows the chemical composition of the EO from *L. camara* corresponding to the highest abundant compounds caryophyllene (12.2%), 3-elemene (8.89%), Germacrene D (5.46%),1,2-cyclopentanedione (3.73%), 5-hydroxymethylfurfural (3.57%), 2,3-dihydro-benzofuran (coumaran) (3.53%), geranyl vinyl ether (2.98%), 2,4-dihydroxy-2,5-dimethyl-3(2H)-furan-3-one (2.97%), adipic acid dimethyl ester (2.73%), 3,7-dimethyl-2,3-epoxy-6-octenol (2.71%), hydroxy-dimethyl ester butanedioic acid (2.69%), 2-furylmethanol (2.47%), 2(5H)-furanone (2.11%), and 1-ethylpentyl acetate (2.02%).

The chemical composition of the EO from *A. camphorata* is shown in Table 6, where the highest abundant compounds were germacrene D-4-ol (20.83%), borneol (19.47%), 1,8-cineole (7.71%), longiverbenone (5.15%), ascaridol (4.42%), camphor (4.16%), cyperotundone (2.86%), cedran-8-ol (2.53%), 7-epi-silphiperfol-5-ene (2.08%), (*E*)-isovalencenol (2.04%), β-dihydroagarofuran (2.03%), (*Z*)-piperitol (1.90%), and α-bisabolol oxide A (1.76%).

Table 4. EO Composition of *Cymbopogon citratus* leaves by GC-MS

RT (min)	Area %	Compound name	Match factor
4.39	2.25	4-Methyl-valeric acid	856
4.94	5.4	2-Furanmethanol	925
5.33	0.24	Edulan II	687
5.47	0.96	2,4-Dimethyl-5-methylthiopent-4-en-2-ol	763
5.82	0.75	5-Aminolevulinic acid	831
6.19	1.91	4-Methoxycoumarin	954
6.85	1.63	2,4-Dihydroxy-2,5-dimethyl-3(2H)-furan-3-one	932
7.27	0.91	(11E)-10-Methyl-11-tridecenyl propionate	635
8.47	2.38	*cis*-Linalool oxide	642
8.79	0.53	Furaneol	684
9.22	0.61	1-Propanol	893
9.36	0.93	10-Methyl-E-11-tridecen-1-ol propionate	601
10.77	8.85	Nerylacetal	780
10.98	0.39	Methyl (6*E*,9*E*,12*E*)-6,9,12-octadecatrienoate	655
12.14	0.29	2-(12-Pentadecynyloxy)tetrahydro-2H-pyran	726
12.46	0.76	4-Methyl itaconate	767
12.57	0.23	7-(2-Methoxyethyl)-2-oxepanone	704
12.80	1.45	2,3-Dihydro-benzofuran	894
13.72	0.34	Geranyl vinyl ether	760
14.75	8.45	Neral	764
14.96	1.18	(2*E*,3*Z*)-2-Ethylidene-6-methyl-3,5-heptadienal	877
15.11	1.10	13,16-Octadecadienoic acid, methyl ester	696
16.01	3.92	10-Hydroxygeraniol	915
16.22	2.19	Sobrerol-8-acetate	830
17.15	3.85	*cis*-Verbenol	800
17.41	0.25	Ascaridole	785
18.60	0.23	1-Methyl n-l-alpha-aspartyl-l-phenylalanate	700
19.00	1.33	6-Methyl-2-(2-oxiranyl)-5-hepten-2-ol	716
19.84	6.36	Linalool oxide	783
19.96	0.81	2,4-Dimethylhexan-3-ol	725
20.31	25.95	Geranial	734
21.02	4.29	*cis*-Linalool oxide	773
26.03	0.86	9-Thiabicyclo[3.3.1]nonane-2,6-dione	693
29.43	0.29	Limonen-6-ol, pivalate	784
29.90	0.47	*n*-Hexadecanoic acid	855
34.32	1.20	9-(3,3-Dimethyloxiran-2-yl)-2,7-dimethylnona-2,6-dien-1-ol	760

Table 5. EO Composition from *Lantana camara* Leaves

RT (min.)	Area %	Compound	Match Factor
4.04	0.83	3-Methylcyclopentan-1,2-diol	686
4.16	0.39	*trans*-2-Undecenoic acid	693
4.26	0.16	(*E*)-4-Nonenal	790
4.48	1.39	7-Nonenoic-7,8-D2 acid, methyl ester	703
4.72	2.47	2-Furylmethanol	826
5.23	1.39	2,4-Dimethyl-5-methylthiopent-4-en-2-ol	758
5.42	0.59	2-Mercapto-1-hexanol	736
5.58	2.11	2(5H)-Furanone	875
5.72	0.77	3-Nonynoic acid	634

5.81	3.73	1,2-Cyclopentanedione	879
6.05	2.71	3,7-Dimethyl-2,3-epoxy-6-octenol	801
6.67	2.97	2,4-Dihydroxy-2,5-dimethyl-3(2H)-furan-3-one	951
6.76	0.16	2-(2-Methyl-2-Propenyloxy)Ethanol	710
7.41	1.43	*p*-Menth-8-en-2-ol	843
7.72	0.38	4-Isopropyl-1,3-cyclohexanedione	725
8.84	1.55	Furaneol	705
8.97	0.90	1,2-Diol-7-Octene	640
9.25	0.25	*β*-Hydroxymyristic acid	663
9.35	0.38	1-Butoxy-2-ethylhexane	731
9.50	1.34	2,3-Dimethylfumaric acid	794
9.61	0.57	Fumaric acid, 3-methylbut-3-enylundecyl ester	713
10.13	2.69	Hydroxy-dimethyl ester butanedioic acid	805
10.50	0.71	4-Amino-1,5-pentandioic acid	675
10.86	12.2	Caryophyllene	806
11.76	0.74	1,5-Di-O-benzoyl-2-deoxypentofuranose	700
11.93	0.65	3,5-Dihydroxy-2-methyl-4-pyrone	794
12.13	0.76	2-(12-Pentadecynyloxy)tetrahydro-2H-pyran	728
12.22	1.17	Valeric acid, dodecyl ester	683
12.58	0.15	*exo*-2-Hydroxycineole acetate	722
12.78	3.53	2,3-Dihydro-benzofuran (Coumaran)	886
13.13	3.57	5-Hydroxymethylfurfural	867
13.72	2.98	Geranyl vinyl ether	745
14.12	0.72	6-Hydroxy-9-oxabicyclo[3.3.1]nonan-2-one	668
14.95	1.79	(2*E*,3*Z*)-2-Ethylidene-6-methyl-3,5-heptadienal	659
15.29	0.71	Tridecanedial	714
15.35	0.43	(*E*)-10-Heptadecen-8-ynoic acid, methyl ester	698
15.96	1.31	2,6-Dimethoxyphenol	856
16.47	2.02	1-Ethylpentyl acetate	790
16.71	0.17	3-(2-Aminoethyl)-1H-indol-5-ol	684
17.99	0.64	Methyl 4,6-tetradecadiynoate	750
18.33	2.85	1-Methoxy-1,9(*Z*)-octadecadiene	718
18.85	1.47	2,7-Dioxa-tricyclo[4.4.0.0(3,8)]deca-4,9-diene	724
20.86	2.73	Adipic acid dimethyl ester	891
22.06	0.70	2-Butyl-4-isopropyl-2-cyclohexen-1-one	817
22.22	0.34	Oleic acid	756
22.63	0.42	12,15-Octadecadiynoic acid, methyl ester	749
22.82	0.89	3-Hydroxydodecanoic acid	738
22.92	0.78	1-Acetyl-19,21-epoxy-15,16-dimethoxy-aspidospermidin-17-ol	761
23.22	0.60	*cis*-1,2-Cyclododecanediol	740
24.32	8.89	3-Elemene	869
25.39	5.46	Germacrene D	827
25.88	1.74	Thujopsenal	776
27.19	0.79	9-(3,3-Dimethyloxiran-2-yl)-2,7-dimethylnona-2,6-dien-1-ol	795
28.66	0.30	*E,E,Z*-1,3,12-Nonadecatriene-5,14-diol	750
29.37	0.77	Nerolidol-epoxyacetate	842
29.90	0.95	Palmitic acid	879
33.11	0.21	Methyl hexadecadienoate	829
33.21	0.70	Octadec-9-enoic acid	854
33.65	0.22	2,3-Dihydroxypropyl palmitate	784

Table 6. EO Composition from *Artemisia camphorata* Leaves

RT	Area %	Compound	Match Factor
7.81	1.38	*α*-Terpinene	910
8.89	7.71	1,8-Cineole	755
9.93	1.44	Artemisia ketone	898
11.04	0.38	4-Thujanol	886
11.65	0.56	*trans-para*-2-Menthen-1-ol	873
14.11	19.47	Borneol	878
14.73	1.33	*trans*-Piperitol	881
14.91	1.90	(*Z*)-Piperitol	833
15.47	1.35	Silphiperfol-5-ene	874
16.13	2.08	7-epi-Silphiperfol-5-ene	866
16.44	0.79	Bornyl acetate	891
17.12	2.04	(*E*)-Isovalencenol	686
17.68	0.71	*α*-Patchoulene	787
18.95	0.64	Isolongifolol, acetate	745
19.26	4.42	Ascaridol	800
19.95	0.86	Aromadendrene	856
20.83	4.16	Camphor	918
21.83	0.89	Guaia-3,9-diene	788
22.55	2.53	Cedran-8-ol	755
23.57	2.03	*β*-Dihydroagarofuran	751
24.31	0.99	24-Norursa-3,12-diene	723
25.52	2.86	Cyperotundone	711
25.77	0.22	9-Methoxy-2,3-dihydrofuro[3,2-g]coumarin	872
26.29	20.83	Germacrene D-4-ol	869
26.58	5.15	Longiverbenone	764
26.78	0.30	Isoaromadendrene epoxide	753
27.12	0.76	*γ*-Himachalene	740
27.57	1.51	11,11-Dimethyl-4,8-dimethylenebicyclo[7.2.0]undecan-3-ol	894
28.13	1.38	*α*-Bisabolol	892
28.68	1.02	*α*-Santalol	721
28.95	1.03	Bisabolone oxide A	917
29.54	0.28	*α*-Cedrene epoxide	904
29.72	0.18	Liguhodgsonal	867
30.86	1.76	*α*-Bisabolol oxide A	902
32.62	1.01	1-Hydroxy-α-bisabolol oxide A acetate	901
32.74	0.26	2-Acetyl-4-(2,6-dimethylphenyl)-2h-1,4-thiazin-3(4H)-one	844
33.62	1.46	2-Butenoic acid, 4-(2-pentyl-1,3-dioxolan-2-yl)-, ethyl ester	956
36.65	0.23	8,9,10,11-Tetrahydrobenzo[a]anthracene	901
39.42	0.21	5-Hydroxymethyl-1,3,3-trimethyl-2-(3-methyl-buta-1,3-dienyl)-cyclopentanol	681

Table 7 presents the chemical compounds of *I. cylindrica* leaf HOE, where the highest abundant compounds were 5-phenylundecane (10.68%), 6-phenyldodecane (8.70%), 2-phenylundecane (5.84%), 4-phenylundecane (5.15%), 3-phenylundecane (5.08%), 2-phenyldodecane (4.98%), 3-phenyldodecane (4.65%), 6-ethyl-5-hydroxy-2,3,7-trimethoxynaphthoquinone (4.34%), 5-phenyldecane (4.31%), 6-phenyltridecane (4.09%), 4-phenyldecane (3.84%), 3-phenyldecane (3.76%), 2-phenyldecane (3.47%), 2-phenyltridecane (3.24%), 4-phenyltridecane (3.25%), and 3-phenyltridecane (3.13%).

Table 7. EO Composition from of *Imperata cylindrica* Leaves

RT	Area %	Compound	Match Factor
14.28	0.15	1-Heptadecene	772
14.50	1.56	Tridecane	945
16.89	0.44	Cyclotetradecane	916
20.12	0.90	3,4-Dihydro-2H-1,5-(3"-t-butyl)benzodioxepine	945
20.62	4.31	5-Phenyldecane	904
20.84	3.84	4-Phenyldecane	910
21.25	3.76	3-Phenyldecane	899
22.11	3.47	2-Phenyldecane	899
22.96	10.68	5-Phenylundecane	886
23.24	5.15	4-Phenylundecane	915
23.45	0.32	4-phenyldodecane	778
23.70	5.08	3-Phenylundecane	920
24.51	5.84	2-Phenylundecane	916
24.75	0.32	(1-methyl-1-propylpentyl)benzene	842
25.13	8.70	6-Phenyldodecane	920
25.94	4.65	3-Phenyldodecane	908
26.18	0.12	Isomeric dodecylbenzene	768
26.46	0.87	1-Docosene	949
26.75	4.98	2-Phenyldodecane	925
27.20	4.09	6-Phenyltridecane	878
27.39	2.46	5-phenyltridecane	904
27.62	3.25	4-Phenyltridecane	871
28.06	3.13	3-Phenyltridecane	875
28.83	3.24	2-Phenyltridecane	923
29.45	4.34	6-Ethyl-5-hydroxy-2,3,7-trimethoxynaphthoquinone	947
29.55	0.16	4-phenyleicosane	767
30.11	1.09	*n*-Hexadecanoic acid	917
30.78	0.15	2-phenyltetradecane	822
32.71	1.79	Phytol	926
33.22	0.11	1-Heptatriacotanol	818
34.12	0.26	Heptacos-1-ene	933
39.99	0.37	Diisooctyl phthalate	958
42.33	0.16	Heptacosane	906
43.78	0.11	2-(Tetradecyloxy)ethyl palmitate	750
44.13	0.16	Dotriacontane	824

Among botanical extracts, oil extracts have received much attention as pest control agents (Campolo *et al.* 2018). They are valuable characterized by less persistence in air, low toxicity to human and animals, high volatility, and toxicity to insect pests of stored products (Batish *et al.* 2008).

In the present work, essential oil from *L. camara* showed the highest potential insecticidal activity against the adults of *S. oryzae*, followed by *C. citratus*, *A. camphorata*, and *I. cylindrica* with LC_{50} values of 9.81 mg/cm^2, 10.89 mg/cm^2, 16.12 mg/cm^2, and 36.85 mg/cm^2, respectively.

Oil extracted from Cymbopogon species have shown potential repellent activity against storage insect pests, *S. oryzae*, *Tribolium castaneum*, *Oryzaephilus surinamensis*, and *Sitophilus zeamais* (Saljoqi *et al.* 2006; Olivero-Verbel *et al.* 2010; Hernandez-Lambraño *et al.* 2015). By GC-MS, β-citral (43.63%) and geranial (41.51%), were the highest abundant compounds in the oil from *C. citratus* grown in Egypt (Mansour *et al.* 2020); also geranial (42.2%) and neral (31.5%), were the main compounds of the oil from

Algerian *C. citratus* (Boukhatem *et al.* 2014), and from Brazil, where geranial (*α*-citral) and neral were main compounds (Silva *et al.* 2008). In the present study, germacrene D-4-ol, borneol, 1,8-cineole, longiverbenone, ascaridol, and camphor were the highest abundant co compounds in *A. camphorata* EO. *A. camphorata* showed the major compounds germacrene D-4-ol, 1,8-cineole, ascaridole and borneol with percentages of 22, 12, 10, and 10 %, respectively in Brazilian plant (Seixas *et al.* 2018). *A. camphorata* EO showed the presence of davanone (20%) as major component in Tunisia plant (Mohsen and Ali 2009).

The most interesting finding in this study is the potent contact toxicity of *Lantana camara* oil against the rice weevil, *S. oryzae* followed by *C. citratus* oil then ending with *A. camphorata* and *Imperata cylindrica* oil by residual film technique. These findings were in accordance with the results of Morya *et al.* (2010), which demonstrated the efficacy of *L. camara* (L.) powdered leaves against the stored grain insect pest *Corcyra cephalonica* (Stainton). Also, *L. camara* hexane extract could be used as protectant against maize weevil, *S. zeamais* Motsch. and bean weevil, *Callosobruchus maculatus* (F.) (Ogunsina *et al.* 2011).

Both *C. citratus* powder and methanol extract were promised as insecticides and can be used effectively in the management of *S. oryzae* in storage (Uwamose *et al.* 2017). The powdered leaves of Spear grass (*I. cylindrica*) could serve as a safer alternative to synthetic insecticides for the maize weevil (*S. zeamais*) infestation in stored maize grains (Ehisianya *et al.* 2019). Extracts from *L. camara* leaf were used for the stored maize protection against the infestation of *S. zeamais* (Ayalew 2020). Recently, Loko *et al*. (Loko *et al.* 2021) showed the good potential of *C. citratus* as both antifeedant and fumigant toxic agent against *Dinoderus porcellus* Lesne.

Furthermore, Stefanazzi *et al*. (2011) stated that *L. camara* and *C. citratus* oils can be used in IPM programmes for *S. oryzae* control. Bhatt *et al*. (2014) showed the toxicity and the significant reduction in larval weight of 2nd and 6th day's old larvae of *Spodoptera litura* as affected by *L. camara* (fruits). *L. camara* leaf extracts can be useful as a biorotional insecticide against *Cadra cautella* (Walker) eggs, larvae and adults (Gotyal *et al.* 2016). Furthermore, the aqueous extract of *C. citratus* Stapf leaf extract gave the highest protection on the maize grains against *S. zeamais* Motsch (Oboho *et al.* 2017).

Taxodium oil from trees grown in El-Beheira, Egypt, with its main compounds of α-pinene, thujopsene, and cedrol followed by carvone and oils from trees grown in Alexandria, Egypt, with its main compounds germacrene D (14.71%), borneol acetate (6.81%), ledene oxide-(II) (6.13%), and *trans*(*β*)-caryophyllene (6.13%) showed the potential LC_{50} against *S. oryzae* with values of <50, 28.52, and>50 μL/L, respectively (Abdelsalam *et al.* 2019). Recently, the acetone extract from leaves of peach trees treated with Ag NPs (15 mL/L) showed LC_{50} of 955.24 ppm against the rice weevils *S. oryzae* (Mosa *et al.* 2021). Essential oils from *A. camphorata*, *A. absinthium*, *A. annua*, *A. dracunculus*, and *A. vulgaris* showed potential activity against the melonworm *Diaphania hyalinata* (Linnaeus 1758) (Lepidoptera: Crambidae) larvae with different levels (Seixas *et al.* 2018).

The inhibitory effects of four tested oils on acetylcholinesterase (AChE) and alkaline phosphatase (ALP) activities in *S. oryzae* were examined *in vivo*. Several extracted oils have been described as inhibitors of AChE and ALP isolated from *S. oryzae* and other insect species (Kim *et al.* 2013; Abdelgaleil *et al.* 2016; Oboh *et al.* 2017). The inhibition of AChE activity may lead to the accumulation of acetylcholine at neuromuscular junctions, which ultimately led to insect mortality (Rajashekar *et al.* 2014).

Transaminases link the metabolism of amino acids, lipids, and carbohydrates. Exposure to the extracted oils caused significant decrease in the AST and ALT activities in *S. oryzae* adults. A similar pattern of reduction in AST (14.29%) and ALT (6.98%) level was observed when cotton leafworm, *S. littoralis*, was exposed to spinosad (Abd El-Mageed and Elgohary 2006).

From this perspective, the present findings could increase demand and experience in the use of these eco-friendly natural products, which could replace synthetic chemicals for management stored grain protection. Finally, these oils are recommended to be used as eco-friendly natural products and could replace synthetic chemicals for management stored grain protection.

CONCLUSIONS

1. *Cymbopogon citratus*, *Lantana camara*, *Artemisia camphorate*, and *Imperata cylindrica* oil extracts showed insecticidal potency against the adults of *Sitophilus oryzae*.
2. In terms of the enzymes inhibition percentages, *L. camara* essential oil gave the best results with AChE, ALP, AST, and ALT.
3. According to contact toxicity of the oils against *S. oryzae* adults after 72 h, *L. camara* EO gave the highest effect followed by *C. citratus* EO.

ACKNOWLEDGMENTS

This research was funded by the Researchers Supporting Project (RSP-2021/123) King Saud University, Riyadh, Saudi Arabia.

REFERENCES CITED

Abbott, W. S. (1925). "A method of computing the effectiveness of an insecticide," *Journal of Economic Entomology* 18(2), 265-267. DOI: 10.1093/jee/18.2.265a

Abd El-Mageed, A., and Elgohary, L. (2006). "Impact of spinosad on some enzymatic activities of the cotton leafworm," *Pakistan Journal of Biological Sciences* 9(4), 713-716. DOI: 10.3923/pjbs.2006.713.716

Abdelgaleil, S. A. M., Mohamed, M. I. E., Shawir, M. S., and Abou-Taleb, H. K. (2016). "Chemical composition, insecticidal and biochemical effects of essential oils of different plant species from Northern Egypt on the rice weevil, *Sitophilus oryzae* L.," *Journal of Pest Science* 89(1), 219-229. DOI: 10.1007/s10340-015-0665-z

Abdelsalam, N. R., Salem, M. Z. M., Ali, H. M., Mackled, M. I., El-Hefny, M., Elshikh, M. S., and Hatamleh, A. A. (2019). "Morphological, biochemical, molecular, and oil toxicity properties of *Taxodium* trees from different locations," *Industrial Crops and Products* 139, 111515. DOI: 10.1016/j.indcrop.2019.111515

Abd-Elkader, D. Y., Salem, M. Z. M., Komeil, D. A., Al-Huqail, A. A., Ali, H. M., Salah, A. H., Akrami, M., and Hassan, H. S. (2021). "Post-harvest enhancing and *Botrytis cinerea* control of strawberry fruits using low cost and eco-friendly natural

oils," *Agronomy* 11, 1246. DOI: 10.3390/agronomy11061246
Ali, H. M., Elgat, W. A. A. A., El-Hefny, M., Salem, M. Z. M., Taha, A. S., Al Farraj, D. A., Elshikh, M. S., Hatamleh, A. A., and Abdel-Salam, E. M. (2021). "New approach for using of *Mentha longifolia* L. and *Citrus reticulata* L. essential oils as wood-biofungicides: GC-MS, SEM, and MNDO quantum chemical studies," *Materials* 14, 1361. DOI: 10.3390/ma14061361.
Arthur, F. H., Campbell, J. F., and Toews, M. D. (2014). "Distribution, abundance, and seasonal patterns of stored product beetles in a commercial food storage facility," *Journal of Stored Products Research* 56, 21-32. DOI: 10.1016/j.jspr.2013.11.003
Ayalew, A. A. (2020). "Insecticidal activity of *Lantana camara* extract oil on controlling maize grain weevils," *Toxicology Research and Application* 4, 2397847320906491. DOI: 10.1177/2397847320906491
Batish, D. R., Singh, H. P., Kohli, R. K., and Kaur, S. (2008). "Eucalyptus essential oil as a natural pesticide," *Forest Ecology and Management* 256(12), 2166-2174. DOI: 10.1016/j.foreco.2008.08.008
Behiry, S. I., Nasser, R. A., Abd El-Kareem, M. S. M., Ali, H. M., and Salem, M. Z. M. (2020). "Mass spectroscopic analysis, MNDO quantum chemical studies and antifungal activity of essential and recovered oil constituents of lemon-scented gum against three common molds," *Processes* 8, 275, DOI: 10.3390/pr8030275
Bhatt, P., Thodsare, N., and Srivastava, R. (2014). "Toxicity of some bioactive medicinal plant extracts to Asian army worm, *Spodoptera litura*," *Journal of Applied and Natural Science* 6(1), 139-143. DOI: 10.31018/jans.v6i1.390
Boukhatem, M. N., Ferhat, M. A., Kameli, A., Saidi, F., and Kebir, H. T. (2014). "Lemon grass (*Cymbopogon citratus*) essential oil as a potent anti-inflammatory and antifungal drugs," *Libyan J Med* 9, 25431-25431. DOI: 10.3402/ljm.v9.25431
Campolo, O., Giunti, G., Russo, A., Palmeri, V., and Zappalà, L. (2018). "Essential oils in stored product insect pest control," *Journal of Food Quality* 2018, 6906105. DOI: 10.1155/2018/6906105
CoStat Statistical Software. (2005). *Microcomputer Program Analysis Version 6.311*, CoHort Software, Berkeley, CA, USA.
Dal Bello, G., Padin, S., López Lastra, C., and Fabrizio, M. (2000). "Laboratory evaluation of chemical-biological control of the rice weevil (*Sitophilus oryzae* L.) in stored grains," *Journal of Stored Products Research* 37(1), 77-84. DOI: 10.1016/S0022-474X(00)00009-6
Ehisianya, C., Ikpi, P., Okore, O., and Obeagu, I. (2019). "Assessment of plant powders as protectants of stored maize grains against maize weevil, *Sitophilus zeamais* (Motsch).[Coleoptera: Curculionidae]," *Nigeria Agricultural Journal* 50(1), 37-45.
Ellman, G. L., Courtney, K. D., Andres, V., and Featherstone, R. M. (1961). "A new and rapid colorimetric determination of acetylcholinesterase activity," *Biochemical Pharmacology* 7(2), 88-95. DOI: 10.1016/0006-2952(61)90145-9
El-Sabrout, A. M., Salem, M. Z. M., Bin-Jumah, M., Allam, A. A. (2019). "Toxicological activity of some plant essential oils against *Tribolium castaneum* and *Culex pipiens* larvae," *Processes* 7, 933, DOI: 10.3390/pr7120933
Etebari, K., Mirhoseini, S. Z. and Matindoost, L. (2005). "A study on interaspecific biodiversity of eight groups of silkworm (*Bombyx mori*) by biochemical markers," *Insect Science* 12(2), 87-94. DOI: 10.1111/j.1744-7917.2005.00010.x
Fields, P., and Korunic, Z. (2000). "The effect of grain moisture content and temperature on the efficacy of diatomaceous earths from different geographical locations against

stored-product beetles," *Journal of Stored Products Research* 36(1), 1-13. DOI: 10.1016/S0022-474X(99)00021-1

Gotyal, B. S., Srivastava, C. and Walia, S. (2016). "Toxicity of *Lantana camara* leaf extracts against almond moth, *Cadra cautella* (Walker)," in: *Proceedings of the National Academy of Sciences, India Section B: Biological Sciences* 86(1), 199-204. DOI: 10.1007/s40011-014-0438-0

Hamada, H. M., Awad, M., El-Hefny, M. and Moustafa, M. A. M. (2018). "Insecticidal activity of garlic (*Allium sativum*) and ginger (*Zingiber officinale*) oils on the cotton leafworm, *Spodoptera littoralis* (Boisd.) (Lepidoptera: Noctuidae)," *African Entomology* 26(1) 84-94. DOI: 10.4001/003.026.0084

Hernandez-Lambraño, R., Pajaro-Castro, N., Caballero-Gallardo, K., Stashenko, E. and Olivero-Verbel, J. (2015). "Essential oils from plants of the genus Cymbopogon as natural insecticides to control stored product pests," *Journal of Stored Products Research*, 62, 81-83. DOI: 10.1016/j.jspr.2015.04.004

Isman, M. B. (2005). "Botanical insecticides, deterrents, and repellents in modern agriculture and an increasingly regulated world," *Annual Review of Entomology*, 51(1), 45-66. DOI: 10.1146/annurev.ento.51.110104.151146

Kim, S.-W., Kang, J., and Park, I.-K. (2013). "Fumigant toxicity of Apiaceae essential oils and their constituents against *Sitophilus oryzae* and their acetylcholinesterase inhibitory activity," *Journal of Asia-Pacific Entomology* 16(4), 443-448. DOI: 10.1016/j.aspen.2013.07.002

Klin, Z. (1972). "Recommendations of the German Society for Clinical Chemistry. Standardization of methods for the estimation of enzyme activities in biological fluids. Experimental basis for the optimized standard conditions," *Zeitschrift für Klinische Chemie und Klinische Biochemie* 10(6), 281-91.

Loko, Y. L. E., Medegan Fagla, S., Kassa, P., Ahouansou, C. A., Toffa, J., Glinma, B., Dougnon, V., Koukoui, O., Djogbenou, S. L., Tamò, M., *et al.* (2021). "Bioactivity of essential oils of *Cymbopogon citratus* (DC) Stapf and *Cymbopogon nardus* (L.) W. Watson from Benin against *Dinoderus porcellus* Lesne (Coleoptera: Bostrichidae) infesting yam chips," *International Journal of Tropical Insect Science* 41(1), 511-524. DOI: 10.1007/s42690-020-00235-3

Lowry, O. H., Rosebrough, N. J., Farr, A.L. and Randall, R. J. (1951). "Protein measurement with the Folin phenol reagent," *Journal of Biological Chemistry* 193, 265-275.

Mansour, M. M. A., El-Hefny, M., Salem, M. Z. M., and Ali, H. M. (2020). "The biofungicide activity of some plant essential oils for the cleaner production of model linen fibers similar to those used in ancient Egyptian mummification," *Processes* 8(1), 79. DOI: 10.3390/pr8010079

Miao, Y. G. (2002). "Studies on the activity of the alkaline phosphatase in the midgut of infected silkworm, *Bombyx mori* L.," *Journal of Applied Entomology* 126(2-3), 138-142. DOI: 10.1046/j.1439-0418.2002.00625.x

Mohamed, A. A., Behiry, S. I., Ali, H. M., El-Hefny, M., Salem, M. Z. M., and Ashmawy, N. A. (2020). "Phytochemical compounds of branches from *P. halepensis* oily liquid extract and *S. terebinthifolius* essential oil and their potential antifungal activity," *Processes* 8(3), 330. DOI: 10.3390/pr8030330

Mohsen, H., and Ali, F. (2009). "Essential oil composition of *Artemisia herba-alba* from southern Tunisia," *Molecules* 14(4), 1585-1594. DOI: 10.3390/molecules14041585

Morya, K., Pillai, S. and Patel, P. (2010). "Effect of powdered leaves of *Lantana camara*, *Clerodendrum inerme* and *Citrus limon* on the rice moth, *Corcyra cephalonica*," *Bulletin of Insectology* 63(2), 183-189.

Mosa, W. F. A., El-Shehawi, A. M., Mackled, M. I., Salem, M. Z. M., Ghareeb, R. Y., Hafez, E. E., Behiry, S. I., and Abdelsalam, N. R. (2021). "Productivity performance of peach trees, insecticidal and antibacterial bioactivities of leaf extracts as affected by nanofertilizers foliar application," *Scientific Reports* 11(1), 10205. DOI: 10.1038/s41598-021-89885-y

Moustafa, M. A. M., Awad, M., Amer, A., Hassan, N. N., Ibrahim, E. D. S., Ali, H. M., Akrami, M., and Salem, M. Z. M. (2021). "Insecticidal activity of lemongrass essential oil as an eco-friendly agent against the black cutworm *Agrotis ipsilon* (Lepidoptera: Noctuidae)," *Insects* 12(8), 737. DOI: 10.3390/insects12080737

Neethirajan, S., Karunakaran, C., Jayas, D. S. and White, N. D. G. (2007). "Detection techniques for stored-product insects in grain," *Food Control* 18(2), 157-162. DOI: 10.1016/j.foodcont.2005.09.008

Oboh, G., Ademosun, A. O., Olumuyiwa, T. A., Olasehinde, T. A., Ademiluyi, A. O., Adeyemo, A. C. (2017). "Insecticidal activity of essential oil from orange peels (*Citrus sinensis*) against *Tribolium confusum*, *Callosobruchus maculatus* and Sitophilus oryzae and its inhibitory effects on acetylcholinesterase and Na^+/K^+-ATPase activities," *Phytoparasitica* 45(4), 501-508. DOI: 10.18311/jbc/2016/15540

Oboho, D., Eyo, J., Ekeh, F., Okweche, S. (2017). "Efficacy of *Cymbopogon citratus* Stapf leaf extract as seed protectant against *Sitophilus zeamais* Motschulsky (Coleoptera: Curculionidae) on stored maize (*Zea mays* L.)," *Journal of Biological Control* 30, 220-225, DOI: 10.18311/jbc/2016/15540.

Ogunsina, O., Oladimeji, M., and Lajide, L. (2011). "Insecticidal action of hexane extracts of three plants against bean weevil, *Callosobruchus maculatus* (F.) and maize weevil, *Sitophilus zeamais* Motsch," *Journal of Ecology and the Natural Environment* 3(1), 23-28. DOI: 10.5897/JENE.9000069

Olivero-Verbel, J., Nerio, L. S., and Stashenko, E. E. (2010). "Bioactivity against *Tribolium castaneum* Herbst (Coleoptera: Tenebrionidae) of *Cymbopogon citratus* and *Eucalyptus citriodora* essential oils grown in Colombia," *Pest Management Science* 66(6), 664-668. DOI: 10.1002/ps.1927

Parugrug, M. L., and Roxas, A. C. (2008). "Insecticidal action of five plants against maize weevil, *Sitophilus zeamais* Motsch. (Coleoptera: Curculionidae)," *Current Applied Science and Technology* 8(1), 24-38.

Finney, D. J. (1952). *Probit Analysis: A Statistical Treatment of the Sigmoid Response Curve*, Cambridge University Press, Cambridge.

Pugazhvendan, S., Elumalai, K., Ross, P. R., and Soundarajan, M. (2009). "Repellent activity of chosen plant species against *Tribolium castaneum*," *World Journal of Zoology* 4(3), 188-190.

Qi, Y.-T., and Burkholder, W. E. (1981). "Protection of stored wheat from the granary weevil by vegetable oils," *Journal of Economic Entomology* 74(5), 502-505. DOI: 10.1093/jee/74.5.502

Rajashekar, Y., Raghavendra, A., and Bakthavatsalam, N. (2014). "Acetylcholinesterase inhibition by biofumigant (coumaran) from leaves of *Lantana camara* in stored grain and household insect pests," *BioMed Research International* 2014, 187019. DOI: 10.1155/2014/187019

Rajendran, S., and Sriranjini, V. (2008). "Plant products as fumigants for stored-product insect control," *Journal of Stored Products Research* 44(2), 126-135. DOI:

10.1016/j.jspr.2007.08.003

Rattan, R. S. (2010). "Mechanism of action of insecticidal secondary metabolites of plant origin," *Crop Protection* 29(9), 913-920. DOI: 10.1016/j.cropro.2010.05.008

Reitman, S., and Frankel, S. (1957). "A colorimetric method for the determination of serum glutamic oxalacetic and glutamic pyruvic transaminases," *American Journal of Clinical Pathology* 28(1), 56-63. DOI: 10.1093/ajcp/28.1.56

Salem, M. Z. M., Ali, H. M., and Basalah, M. O. (2014). "Essential oils from wood, bark, and needles of *Pinus roxburghii* Sarg. from Alexandria, Egypt: Antibacterial and antioxidant activities," *BioResources* 9(4), 7454-7466. DOI: 10.15376/biores.9.4. 7454-7466

Salem, M. Z. M., Ali, M. F., Mansour, M. M. A., Ali, H. M., Abdel Moneim, E. M., Abdel-Megeed, A. (2020). "Anti-termitic activity of three plant extracts, chlorpyrifos, and a bioagent compound (Protecto) against termite *Microcerotermes eugnathus* Silvestri (Blattodea: Termitidae) in Egypt," *Insects* 2020, 11, 756. DOI: 10.3390/insects11110756.

Salem, M. Z. M., El-Hefny, M., Ali, H. M., Abdel-Megeed, A., El-Settawy, A. A. A., Böhm, M., Mansour, M. M. A., and Salem, A. Z. M. (2021). "Plants-derived bioactives: Novel utilization as antimicrobial, antioxidant and phytoreducing agents for the biosynthesis of metallic nanoparticles," *Microbial Pathogenesis* 158, 105107. DOI: 10.1016/j.micpath.2021.105107

Salem, M. Z. M., Behiry, S. I., and EL-Hefny, M. (2019). "Inhibition of *Fusarium culmorum*, *Penicillium chrysogenum* and *Rhizoctonia solani* by n-hexane extracts of three plant species as a wood-treated oil fungicide," *Journal of Applied Microbiology* 126(6), 1683-1699. DOI: 10.1111/jam.14256

Saljoqi, A. U. R., Afridi, M. K., Khan, S. A., and Rehman, S. (2006). "Effects of six plant extracts on rice weevil *Sitophilus oryzae* L. in the stored wheat grains," *Journal of Agricultural and Biological Science* 1(4), 1-5.

Seixas, P. T. L., Demuner, A. J., Alvarenga, E. S., Barbosa, L. C. A., Marques, A., Farias, E. D .S., and Picanço, M. C. (2018). "Bioactivity of essential oils from Artemisia against *Diaphania hyalinata* and its selectivity to beneficial insects," *Scientia Agricola* 75, 519-525. DOI: 10.1590/1678-992X-2016-0461

Silva, C. d. B. d., Guterres, S. S., Weisheimer, V., and Schapoval, E. E. (2008). "Antifungal activity of the lemongrass oil and citral against *Candida* spp.," *Brazilian Journal of Infectious Diseases* 12(1), 63-66.

Solomon, B., Gebre-Mariam, T., and Asres, K. (2012). "Mosquito repellent actions of the essential oils of *Cymbopogon citratus*, *Cymbopogon nardus* and *Eucalyptus citriodora*: Evaluation and formulation studies," *Journal of Essential Oil Bearing Plants* 15(5), 766-773. DOI: 10.1080/0972060X.2012.10644118

Stefanazzi, N., Stadler, T., and Ferrero, A. (2011). "Composition and toxic, repellent and feeding deterrent activity of essential oils against the stored-grain pests *Tribolium castaneum* (Coleoptera: Tenebrionidae) and *Sitophilus oryzae* (Coleoptera: Curculionidae)," *Pest Management Science* 67(6), 639-646. DOI: 10.1002/ps.2102

Tawfeek, M., Abu-Shall, A., Gad, A., and Mohey, M. (2017). "Evaluation of six plant essential oils against three stored product insects and their effects on the Haemogram under laboratory conditions," *Alexandria Journal of Agricultural Sciences* 62(3), 291-301. DOI: 10.21608/alexja.2017.5793

Uwamose, M. O., Nmor, J. C., Okulogbo, B. C., and Ake, J. E. (2017). "Toxicity of lemon grass *Cymbopogon citratus* powder and methanol extract against rice weevil

Sitophilus oryzae (Coleoptera: Curculionidae),” *Journal of Coastal Life Medicine* 5(3), 99-103.
Zibaee, A., Sendi, J. J., Etebari, K., Alinia, F., and Ghadamyari, M. (2008). “The effect of diazinon on some biochemical characteristics of *Chilo suppressalis* Walker (Lepidoptera: Pyralidae), rice striped stem borer,” *Mun. Entomol. Zool*. 3(1), 255-265.
Zibaee, A. (2011). “Botanical insecticides and their effects on insect biochemistry and immunity,” in: *Pesticides in the Modern World - Pests Control and Pesticides Exposure and Toxicity Assessment*, M. Stoytcheva (ed.), InTech, pp. 55-68.

Article submitted: August 31, 2021; Peer review completed: September 28, 2021; Revised version received and accepted: September 29, 2021; Published: October 4, 2021.
DOI: 10.15376/biores.16.4.7767-7783