Egypt. J. Plant Prot. Res. Inst. (2020), 3 (1): 268 - 280

Egyptian Journal of Plant Protection Research Institute

www.ejppri.eg.net

Bioactivity and chemical composition of anise (*Pimpinella anisum*) on *Rhyzopertha dominica* (Coleoptera: Bostrichidae) compared to malathion.

Zayed, G.M.M.[1] and Abou-Elkassem, S.A.A.[2]and Nahed, A. Hasan[3]

[1] *Plant Protection Research Institute, Agricultural Research Center, Dokki, Giza, Egypt.*

[2]*Faculty of Agriculture, Al-Azhar University, Assiut.*

[3]*Pesticide chemistry and Toxicology Department, Faculty of Agriculture, Kafr El-Sheikh University.*

ARTICLE INFO

Article History

Received: 30/ 1/ 2020

Accepted: 22/3 /2020

Keywords

Essential oils, Lesser grain borer, *Rizopertha dominica*, anise (*Pimpinella anisum*) and malathion.

Abstract:

In recent years, the focus has been on using alternatives to control stored grain pests to overcome resistance to pesticides. It reduced the costs, providing good control and safe to use. Ways to protect grain storage include the use of safe alternatives such as plant products. Lesser grain borer, *Rhyzopertha dominica* (Fabricius) (Coleoptera: Bostrichidae) is one of the most important insect pests of stored products in the world. The adult emergence may spend several days within the grain attacking wheat grains causing before making exit holes to emerge. This cause weight loss and reduced nutrition values. The present study was conducted in laboratory to evaluate the efficacy of anise (oil and powder) compared to malathion as a recommended insecticide for controlling stored grain pests. The tested compounds evaluated by mixing with medium assay to study the parameters of mortality %, emergence % of *R. dominica* adults, on the repellent activity, germination %, weight loss % beside the identification of chemical components of anise oil by GC/MS analysis. The results showed that anise oil and powder and malathion were effective in reducing insect infestations. The tested materials significantly increased the insect mortality % and reduced the emergence % with increasing concentrations and exposure time. Also, the weight loss % of wheat grains decreased with increasing concentrations of all tested compounds compared to control. Moreover, the tested materials showed a good repellent activity on *R. dominica* adults especially at the highest concentration. The effect of anise oil was higher than that of the powder and malathion for repellent activity. Furthermore, anise oil slight inhibited the germination percentage of wheat grains followed by anise powder. While, malathion had no effect on germination percentage compared with control, after three months post treatment. Considering the results of current study, it could be suggested that the anise oil and powder have the potentiality to be used as an alternative to chemical insecticides for protecting stored grains against *R. dominica* in the integrated pest management program.

Introduction

Storage of grains is part of the post-harvest system through which food material passes on its way from field to consumer. It is generally accepted that 5–15% of the total weight of all cereals, oil seeds, and pulses is lost after harvest (Anonymous, 1989). Cereals are the staple and nutritive food but their storage is not safe due to the attack of certain stored grain insect pests. Insect infestation alone has been noted for the causes of over 5-10% losses of stored grains in the temperate countries and 20-30% in the tropical zones (Dubey *et al*., 2008).

Lesser grain borer *Rhyzopertha dominica* (Fabricius) (Coleoptera: Bostrichidae) is one of the most important insect pests of stored products in the world. Newly emerging adults may spend several days within the grain before chewing exit holes to emerge (Benhalima *et al*., 2004). Feeding by *R. dominica* larvae and adult can reduce weight by as much as 75% (Dal bello *et al*., 2001),and also reduce nutritional and aesthetic value of the grain. Moreover, the lesser grain borer reduce germination (Moino *et al.,* 1998). Controlling of stored product insect populations is primarily depended upon continued applications of insecticides. However, the implications of these are serious problems of toxic residues, health and environmental hazards, development of insect strain resistant to insecticides, increasing cost of application (Sighamony *et al*., 1986; Okonkwo and Ewete, 1999 and Dubey *et al*., 2008). Malathion-resistant phenotype has almost completely replaced the susceptible strain. Moreover, many of the stored product insects have developed resistance to the commonly used chemicals (Subramanyam and Hagstrum, 1995 and Srivastava and Singh, 2002).

The use of plant oils for controlling insect pests in stored grains is a sustainable alternative because the oils are derived from natural resources. Such oils could function as contact toxin, fumigant, repellent, antifeedant and oviposition inhibitor (Tapondjou *et al*., 2002 and Isman, 2008).

Furthermore, the use of plant materials as traditional protectants of stored products is an old practice used allover the world (Aslam *et al*., 2002). The protection of stored products generally involves mixing grain with plant-based protectants (Tapondjou *et al*., 2002 and Udo *et al*., 2011). It is an age-old practice of traditional farmers in the tropics to mix a local plant with seeds of legumes. Using plant with insecticidal properties is therefore an attractive alternative to the more explosive synthetic insecticides. Various plant byproducts have been tried recently with a good degree of success as protectants against number of stored grain insect pests (Ketoh *et al*., 2005; Hosny *et al*., 2007; Ziga *et al*., 2012 and Wanida *et al*., 2012). Therefore, the goal of this study to evaluate the efficiency of anise oil and powder compared to the recommended compound malathion against *R. dominica* with the respect to adult mortality and progeny reduction, to identify the chemical components of anise oil and also losses of grain weight, repellency and germination of wheat grains has been evaluated.

Materials and methods

1.Materials:

1.1.Tested insects:

Lesser grain borer, *R. dominica* used in this study was reared free of insecticidal contamination at 28 ± 2^{oc},

70±5±R.H.% of the laboratory of Stored Product Pests Research Department, Plant Protection Research Institute, Sakha Agricultural Research Station. The culture was maintained under the same conditions, insects were to emplace in glass jar (1000 gm) containing 500 g of sterilized wheat grain and 400-500 of *R. dominica* insects. Adult insects were left for two weeks for eggs laying in the jar and kept again at the untreated conditions in the rearing laboratory. The newly emerging adults (1-2 weeks-old) of *R. dominica* were used for experimental work.

1.2.The stored product:

Wheat grains used were Masr 1, which obtained from Sakha Agriculture Research Station Farm. The grains were used to culture *R. dominica* and to evaluate the efficacy of anise oil and powder against *R. dominica* compared to malathion insecticide.

1.3.Collection and preparation of plant powder:

The plant powders used (*Pimpinella anisum*) anise seeds were collected from local market. The target plant seeds were dried at the room temperature (25-28^{oc}), the dried seedes powdered mechanically by using an electric blender, then sieved through 300 mesh size. The resulting fine powders were maintained in tightly closed dry bags until used for the experimental work.

1.4.Plant Oil used:

The oil of anise was obtained from Hashem Brothers Company for Essential Oils and Aromatic Products (Kafr Elsohbya, Qalyoubeya, Egypt).

1.5. Analysis of anise essential oil:

The constituents of anise plant oil was analyzed by gas chromatography-mass spectrometry (GC/MS) using HP5890 system with HP column (60 meter x 0.25 millimeter, 0.25 m film thickness). Detector was flame ionization detector (FID). The mobile phase was nitrogen and hydrogen was the stationary phase. Initial temperature was 60^{oc} and maximum temperature was 250^{oc}. The injector temperature was 240^{oc}. Relative percentage amounts were calculated from peaks total area by apparatus software. The compounds were identified by matching the mass spectra data with those held in a computer library (Wiley 275 L). All steps of sample preparation, extraction and analysis procedure were carried out in the Analysis Laboratory of Hashem Brothers for Essential oils and Aromatic Products, Abdel-Moneim Riad St., Giza, Egypt.

1.6.The chemical insecticide (Malathion):

Chemical name: O,O dimethyl-S-(1,2 dicarboxy-ethyl) ethyl phosphorodithioate

The applied formulation: odorless malathion (dust 1%)

Source: Kafr El-Zayat Pesticides and Chemical Company, Egypt

2.Methods:

2.1.Toxicity activity of tested materials against *Rhizopertha dominica* adults:

Mixing with feeding medium technique was used to determine the insecticidal effects of anise oil and powder and malathion against *R. dominica*. The considerable concentrations used were (1.0, 2.0, 3.0 and 4.0%) w/w for anise oil, (0.5, 1.5, 3.0 and 5.0% w/w) for anise powder and (0.04, 0.06, 0.08 and 0.1% w/w) for malathion insecticide. These concentrations of each tested materials were separately mixed with 20 g of wheat grains and were introduced in 250 ml and the jar was shaken hand to mix the grain with all tested concentrations. The jars without any tested materials were used as

control. Each concentration and untreated control replicated three times. Twenty of newly emerged adults of *R. dominica* (1-2 weeks old) were added to each jar, the jars covered with muslin cloth and kept under laboratory conditions. Mortality counts were recorded after 7 and 15 days. All results were corrected with Abbott's formula (1925).

Data were then analyzed using Probit analysis Litchfield and Welcoxon (1949), to estimate LC50, slope value and 95% confidence limits (CL).,Toxicity index.

2.2.Biological activity of tested materials against *Rhizopertha dominica*:

The biological effect of anise (oil and powder) and malathion were evaluated after recording mortality. The desirable concentrations of anise (oil, powder) and malathion were (1.0, 2.0, 3.0 and 4.0% w/w) dissolved in acetone (0.5, 1.5, 3.0 and 5.0% w/w) and (0.04, 0.06, 0.08 and 0.1% w/w) for anise oil, marjoram powder and malathion, respectively. Each concentration was applied in three replicates and in each replicate there were 20 g of wheat grains in 250 mL glass jars. For oil the treatment was carried out by adding 1 ml of each concentration to the wheat grains, mixing well and then left in jars for suitable time until the solvent evaporated before using them in experiment. However, for anise powder and malathion dust the treatments were carried out by mixing powder and dust with wheat grains and were shaken thoroughly to ensure uniform coverage by the different treatments. The untreated treatment was used as control and was replicated three times. After that, 20 adults unsexed (1-2 week-old) of *R. dominica* were transferred to the treated wheat grains in glass jars (250 mL) and kept at 28 ± 1^{oc} and 70 ± 5 R.H. according to the method described by El-Lakwah *et al*. (1992). Mortality counts were recorded after 7 and 15 days. Then the adults were sieved out and discarded after twenty days. The newly adult emergence were used to calculate the reduction percentages in *R. dominica* progeny.

$$\text{Reduction \%} = \frac{\text{No. of adults emerged in control}}{\text{No. of adults emerged in treatment}} \times 100$$

2.3. Repellency activity of tested materials against *Rhizopertha dominica*:

The repellency effect of anise (oil and powder) and malathion against *R.dominica* adult was conducted using the modified apparatus according to Helen (1989). It consists of a metallic ring (6 cm diameter x 1 cm height) was placed at the center of Petri-dish (12 cm diameter x 2.5 cm height). Concentrations of anise oil, powder and malathion (1.0, 2.0, 3.0 and 4% w/w), (0.5, 1.5, 3.0 and 5.0% w/w) and (0.04, 0.06, 0.08 and 0.1% w/w) for anise oil, powder and malathion, respectively. anise oil only was dissolved in 1 ml acetone. However, the treatment was carried out by mixing the anise powder and malathion dust with wheat grains and were shaken thoroughly to ensure uniform coverage with the different treatments. The untreated treatment was used as control. The treatments and control were replicated three times. After that, ten grams of treated wheat grains were put inside the metallic ring. Twenty unsexed adults (1-2 weeks-old) of *R. dominica* were released separately at the center of the ring. The Petri dishes were covered and were kept at 28 ± 1^{oc} and 70 ± 5% R.H. Repellency percentage (PR) values were estimated after 6, 12, 24, 48 and 72 hours according to the following equation of Helen (1989).

% Repellency (PR):

$$P.R. = \frac{\text{No. of adults outside ring}}{\text{Total No. of adults used}} x100$$

2.4. Wheat grains weight loss:

The weight loss of wheat grains due to infestation with *Rhizopertha dominica* was determined three months post treatment by sieving the insects from the wheat grains. Three replicates were done for each treatment and control. The weight loss of wheat grains was calculated as dry weight loss according to the equation of Harris and Lindblad (1978):

$$\% = \frac{\text{initial dry weight of seeds - dry seeds weight after three months}}{\text{Initial dry weight of seeds}} x100$$

2.5. Germination test:

The germination tests for anise (oil and powder) and malathion were accomplished on wheat grains of each treatment according to Qi and Burkholder (1981), with slight modification. Sixty wheat grains of each treatment were divided into three replicates, placed in Petri-dishes containing cotton layers (instead of filter paper) soaked with tap water and covered with tissue. Grains germination percentages were recorded four days after treatment after three months post-treatment. % germination percentages were calculated

2.6. Statistical analysis:

The data were statistically analyzed according to Duncan's multiple range test (**Duncan, 1955)** using SPSS software (1995).

Results and discussion

Results obtained in Table (1) showed that malathion was the most effective agent against *R. dominica* followed by oil powder of anise plant with LC_{50} (0.053 and 0.035), (0.930 and 0.721) and (1.521 and 0.924) for malathion, oil and powder of anise plant after one and two weeks, respectively.

Table (1): Toxicity of malathion, anise oil and powder against *Rhizopertha dominica*.

Total materials	7days					15days				
	LC_{50} %	Confidence limits		S.V.	Toxicity index	LC_{50} %	Confidence limits		S.V.	Toxicity index
		Upper	Lower				Upper	Lower		
Malathion	0.053	0.0735	0.0220	1.8	100	0.035	0.0512	0.0331	2.3	100
Anise oil	0.93	0.978	0.526	1.7	5.69	0.721	0.971	0.563	1.9	4.88
Anise powder	1.521	2.751	0.643	1.3	3.48	0.924	1.211	0.533	1.2	3.62

Also, anise oil was more effective than its powder against *R.dominica*. The LC_{50} values of the tested materials were positively correlated with the time of exposure under all treatments, since the LC_{50} values after 7days were higher than this after 15days in the all treatments. Results in Table 1 were in agreement with those of Derbalah and Ahmed (2011) who found that spearmint oil and powder were effective on the mortality percentage of *Sitophilus oryzae* compared to malathion and mortality increased with increasing exposure time and concentrations with the all tested materials.Gonzalez *et al*. (2014) demonstrated that the geranium and bergamot oils had the highest effective on mortality against *T. castaneum* and *R. dominica* . Adel *et al*. (2015) demonstrated that the higher concentration of basil, fennel, and geranium essential oils achieved 100% mortality resulted in contact toxicity against S. *oryzae* and *C. maculates* adults. However, for *S. oryzae* adults only fennel oil exhibited the lowest LC_{50} followed by basil oil. Geranium oil evoked no detectable mortality of *S. oryzae* adults. Fennel oil induced the

highest mortality rate to *C. maculates* followed by geranium, and basil oils. Akunne and Ononye (2015) recorded high mean mortality of adult S. oryzae in the rice grains treated with (10g/20g) *Piper guineense* and *Citrus sinensis* powder.

1.Biological activity of tested materials against *Rhizopertha dominica*:

The results in Table (2) indicated to the differences in the mortality percentages of *R. dominica* between treatments at 7 and 15 days post treatment. Malathion was the most effective followed by oil and powder of anise plant against adult emergence of *R. dominica*.

Table (2): Effect of malation, anise oil and powder on mortality, reduction % in progeny and wheat grain loss % against *Rhizopertha dominica*.

Tested materials	Conc. w/w %	Morality		Mean no. of adult emergence	Reduction in progeny %	Weight loss of wheat grains
		7 days	15 days			
Malathion	0.04	60.0	70.0	55.0	71.0e	10.0d
	0.06	80.0	86.7	47.0	75.2d	7.1e
	0.08	86.7	93.3	32.0	83.2b	5.0f
	0.10	88.3	98.3	8.0	95.7a	2.7g
Anise oil	1.0	45.0	55.0	75.0	60.5g	14.2 c
	2.0	60.0	70.0	66.0	65.3f	10.2d
	3.0	66.7	75.0	54.0	71.6 e	8.1e
	4.0	80.0	90.0	29.0	84.7 b	4.7 f
Anise powder	0.5	41.3	50.0	91.0	52.1i	20.0 b
	1.5	50.0	61.7	80.0	57.9 h	14.0 c
	3.0	66.7	75.0	60.0	68.4f	10.3d
	5.0	75.0	83.3	36.0	81.0c	7.2e
Control				190.0		46.0 a

Moreover, malathion and anise oil were the most effective treatment on progeny of *R. dominica* followed by anise powder with % reduction values of 95.7, 84.7 and 81.0%,at the highest concentration respectively. In this respect anise oil was more effective than anise powder at all concentration levels. In addition, treatments significant reduced the weight loss with increasing the concentrations. The lowest loss of grain weight was found with the highest concentration. Results also demonstrated that malathion was the premier agent for reducing weight loss followed by anise oil and powder with % values of 2.7, 4.7and 7.2%, respectively. Generally, malathion had the highest effect on the all aspects of the present study. Several studies were conducted on the effect of essential oils on biology of stored product insects. Abo-Arab *et al*. (1998) found that *Nigella sativa* oil at level of 16 ml/kg grains completely prevented adult emergence of *S. oryzae.* Similarly, Abd El-Aziz (2011) found that the marjoram essential oil completely prevented emergence adults of *T. castaneum* and *S. oryzae*. Gamal (2016) mentioned that malathion and the tested plant oils reduced adult emergence of *Callosobruchus maculatus*. Norambuena *et al*. (2016) found that the emergence (F1) was reduced reaching maximums of 60% in the case of *S. granarius* and *S. oryzae*, and 36% in *S. zeamais* by the essential oil *Laureliopsis philippiana*.

2.Repellency activity of tested materials against *Rhizopertha dominica*:

Results shown in Table (3) cleared the repellent effect of tested materials at 6, 12, 24, 48 and 72 hours post treatment. The lowest repellent

effect values were recorded with malathion insecticide. In contrast, the repellent effect of anise oil was more effective than anise powder and malathion against *R. dominica* adults. The repellent effect for all tested materials increased with increasing concentration against *R. dominica* adults. Also, data in Table (3) showed that the tested materials exhibited repellent activity at the highest rate ranged between (96.0-100.0) and (84.0-100.0) and (30.0-44.0) percent of repellent for anise oil,powder and malathion, respectively. Results also showed that the repellent effect decreased with the increasing of exposure time. The obtained results did agree with those of Zapata and Smagghe (2010) reported the repellent activity of the elaves and bark of *Laurelia sempervirens* and *Drimys winters* against *T. castaneum*. The oils tested had a very strong repellent activity towards *T. castanneum* when tested in filter paper arena test. After 4 hrs exposure >90% repellency was achieved Lashgari *et al.* (2014) found that repellency effect was increased with increasing concentration and the highest repellency effect was belonged to the highest concentration. Essential oils of *Mentha piperita* and *Cuminum cyminum* caused 61.2 and 66.4 repellency on *T. castaneum*. Meanwhile their effect was found to be 55.2 ad 60.4% repellency on *S. oryzae* at the highest concentration. Norambuena *et al*. (2016) reported that all treatments of the oil *Laureliopsis philippiana* had a repellent effect against adults of *S. oryzae, S. zeamais,* and *S. granaries.*

Table (3): Repellent effect of the malathion, anise oil and powder against *Rhizopertha dominica.*

Materials	Conc. w/w %	% Repellency				
		Hours post treatment				
		6	12	24	48	72
Malathion	0.04	8.7 h	13.0 i	12.0 i	10.0 i	8.6 i
	0.06	22.0 g	24.0 h	26.0 h	21.0 h	20.0 h
	0.08	27.0 f	32.0 g	34.0 g	25.0 g	22.0 g
	0.10	40.0 e	44.0 f	40.0 f	32.0 f	30.0 f
Anise oil	1.0	72.0 c	78.0 d	70.0 e	61.0 e	58.0 e
	2.0	85.0 b	85.0 b	83.0 c	75.0 c	66.0 d
	3.0	100.0 a	100.0 a	95.0 b	90.0 b	77.0 c
	4.0	100.0 a	100.0 a	100.0 a	97.0a	96.0 a
Anise powder	0.5	66.0 d	73.0e	70.0 e	65.0 d	60.0 e
	1.5	73.0c	77.0 d	78.0 d	60.0 e	61.0 e
	3.0	86.0 b	86.0c	84.0 c	70.0 d	66.0 d
	5.0	100.0 a	97.0 b	97.0 b	86.0b	84.0b

3.Identification of chemical components of anise oil:

The chemical composition of essential oils extracted from anise oil (Table, 4) was determined by GC-MS. The Chromatogram profile of anise oil, *P. anisum* was showed in Figure (1), the highest components were trans-anisole (86.74%), estragole (4.08%) and methyl-chavicol (1.68%).

Results

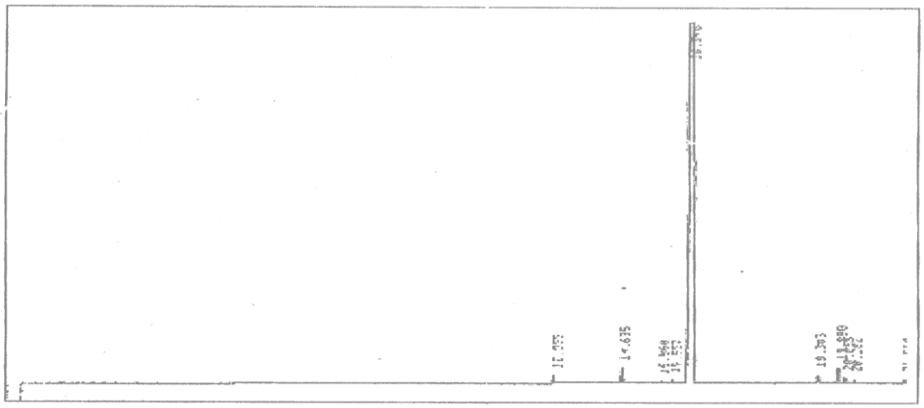

Figure (1): Chromatogram profile of anise seed (*Pimpinella anisum*) essential oil obtained by GC/MS analysis.

Table (4) : Chemical compositions of anise oil, *Pimpinella anisum.*

Main components	Component rate %	Retention time (min.)
Methyl chavicol	1.68	14.61
Trans-anisole	86.74	18.94
Estragol	4.08	25.68

Different studies established the composition of the essential oil of by GC-MS (Özcan and Chalchat (2006) found that the main constituents of *P. anisum* L oil trans-anethole (93.9%) and estragole(2.4%). Soliman *et al*. (2009) reported that the constituents of *O. majorana* L. included terpinen-4-ol (37.4%, 20.5%, 16.3%) was a major component in the summer, autumn and winter oils, resp. and α-terpinene (up to 13.3% in summer). Abd El-Aziz (2011) reported the major constituents of marjoram essential oil plant growing in Egypt are 4-terpineol (29.96%) and β-terpinene (11.34%). Sarrou *et al*. (2013) found that the constituents of *C. aurantium* included limonene (0.53%–94.67%). Ullah *et al*. (2014) reported that the constituents of *P. anisum* L.) oil was trans-anethole (82.1%) . Zarrad *et al*. (2017) found that the constituents of *C. aurantium* included limonene (87.52%).Monoterpenes have insecticidal toxicity including contact and antifeedant action on stored product insect pests. (Lee *et al*., 2003; Rozman *et al*., 2007 and Abdelgaleil *et al*., 2009). The mode of action of bioactivity natural monoterpinoids (hydrocarbons, alcohols and ketones) from spearmint oil may be due to inhibition of acetylcholinesterase. Miyazawa *et al*. (1997) reported that 1, 8-cineole was most potent inhibitor of AChE among the monoterpenes tested. This inhibition may be a mode of action for essential oil and monoterpene. The compounds may prove toxic when penetrating the insect body via the respiratory system (Shaaya *et al*., 1997 and Park *et al*., 2003).

4.Germination tested:

The effect of anise oil and powder and malathion on wheat grains germination percentages after three months post treatment was shown to understand the bioactivities of any essential oil. It is important to know the main chemical composition of the target oil in the research for example all constituents of *C. cyminum* were monoterpenes. In Table (5) the results showed that anise (oil and power) had a slight effect on germination of wheat grains while malathion exhibited non-significant effect on the germination compared to the untreated control.

Table (5): Germination of wheat grains with malation and anise oil and powder after 3 months post-treatment.

Materials	Conc. w/w g/kg	% Germination
Malathion	0.04	100.0 a
	0.06	100.0 a
	0.08	100.0 a
	0.10	100.0 a
Anise oil	1.0	90.0 e
	2.0	86.0 f
	3.0	80.0 g
	4.0	77.0 h
Anise powder	0.5	96.0 b
	1.5	96.0 b
	3.0	94.0 c
	5.0	93.0 d
Control		100.0 a

Anise oil was the highest treatment that reduced germination percentages of wheat grains followed by anise powder. The obtained results agree those of Derbalah and Ahmed (2011) who found that the efficacy of plants evaluated relative to malathion as standard compound to protect wheat against *S. oryzae*. The spearmint oil was the highest treatment that reduced the germination percentage of wheat grains followed by spearmint powder and malathion, respectively. Arya and Tiwari (2013) found that mustard oil at 2% concentration clearly reduced the wheat grains germination.

The current study demonstrated that malathion had distinctive effect on the most investigated parameters compared to anise oil and powder. Since, it achieved the highest effect against *R. dominica* with respect to adult mortality and emergence. However, malathion had many disadvantages serious hazards on human and environment. Furthermore, the development of natural insecticides (anise oil and powder) may help to reduce the negative impact of chemical insecticides malathion because of their low toxicity, no development of resistance of insect and safety to the environment, biodegradable, non-toxic un-target organism, ecofriendly, easily and many plant derived natural products acting against insects could be produced from locally available raw materials. So, the present findings suggest application of anise oil and powder as protectants against the infestation of *R. dominica* as alternatives to the chemical control of *R.dominica*.

References

Abbott, W.S. (1925): A method of the effectiveness of an insecticide. J. Econ. Entomol., 18: 265-279.

Abd El-Aziz, M.f. (2011) : Bioactivities and biochemical effects of marjoram essential oil used against potato tuber moth *Phthorimaea operculella* Zeller (Lepidoptera: Gelechiidae). Life Science J., 8(1): 288-297.

Abdelgaleil, A.; Mohamed, M.; Badaw, M. ; and El-Arami, S. (2009) : Fumigant and contact toxicities of monoterpenes to *Sitophilus zeamais* (L.) and *Tribolium castaneum* (Herbst) and their inhibitory effects on acetylcholinesterase activity. J. Chem. Ecol., 35: 518-525.

Abo-Arab, R.B.; Helal, R.M.Y. and El-Aidy, N.A. (1998) : Bioresidual

activity of certain oils and plant extracts on some stored grain insects in relation with quality of wheat grains. J. Agric. Sci., Mansoura Univ., 23(12): 5635-5641.

Adel, I.; Seada, M. A.; Abo Arab, R. and Seif , A.I. (2015) : Efficacy of three local Egyptian essential oils against the rice weevil, *Sitophilus oryzae* (Coleoptera: Curculionidae) and the cowpea weevil, *Callosobruchus maculatus* (Coleoptera: Bruchidae). Egypt. J. Exp. Biol. (Zool.), 11(1): 95 – 105.

Akunne, C.E. and Ononye, B.U. (2015) : Entomocidal effect of a local spicy and fruit peel powders as rice grain protectant against *Sitophilus oryzae* (L.) Coleoptera: Curculionidae**.** Journal of Entomology and Zoology Studies, 3(3): 208-211.

Anonymous, (1989) : Proceedings of the 18th International Course on Plant Protection. International Agricultural Centre, Wageningen, The Netherlands.

Arya, M. and Tiwari, R. (2013) : Efficacy of plant and animal origin bioproducts against lesser grain borer, *Rhyzopertha dominica* (Fab.) in stored wheat. International J. of Recent Scientific Research, 4(5): 649-653.

Aslam, M.; Ali Khan, Kh. And Bajwa, M.Z.H. (2002) : Potency of some spices against *Callosobruchus chinensis* L. J. Biological Sciences, 2: 449-452.

Benhalima, H.; Choudhary, M.Q.; Millis, K.A. and Price, N. (2004) : Phosphine resistance in stored product insects collected from various grain storage facilities. J. Stored Prod. Res., 40: 241-249.

Dal Bello, G.; Padin, S.; Lopez Lastra, C. and Fabrizio, M. (2001) : Laboratory evaluation of chemical biological control of rice weevil (*Sitophilus oryzae* L.) in stored grains. J. Stored Prod. Res., 37: 77084.

Derbalah, A.S. and Ahmed, S.I. (2011) : Oil and powder of spearmint as an alternative to *Sitophilus oryzae* chemical control of wheat grains. J. Plant Prot. Res., 51: 145-150.

Dubey, N.K.; Srivastava, B. and Kumar, A. (2008) : Current status of plant products as botanical insecticides in storage pest management. J. Biopesti., 1(2): 182-186.

Duncan, D.B. (1955) : Multiple range and multiple F. test. Biometrics, 11: 1-42.

El-Lakwah, F.A.; Darwish, A.A. and Khaled, O. M. (1992) : Effectiveness of dill seed powder (*Anethum graveolens*) on some stored product insects. Ann. Agric. Sci. Moshtohor, 34(4): 2031-2037.

Gamal, M.M. (2016) : Efficacy of three plant oils against the cowpea beetle, *Callosobruchus maculatus* (F.) compared with malathion. Bull. Ent. Soc. Egypt. Econ. Ser., 42: 1530166.

Gonzalez, J.O.W.; Gutierz, M.M.; Ferrero, A.A. and Band, B.F. (2014) : Essential oils nanoformulations for stored product pest control-characterization and biological properties. Chemosphere, 100: 130-138.

Harris, K.L. and Lindblad, C.J. (1978) : Postharvest grain loss assessment method. American Association of Cereal Chemists, St. Paul., Minnesota, USA, pp 193.

Helen, C.F. Su. (1989): Laboratory evaluation of dill seed extracts reducing infestation of rice weevil in stored wheat. J. Entomol. Sci., 24(3): 317-320.

Hosny, A.H.; Salama, A.A.; Ismail, R.; Abo-Arab, R. and Ibrahim, S.I.A. (2007) : Botanical products as stored grain protectants. J. Agric. Res., Kafrelsheikh Univ., 33(4): 873-886.

Isman, M.B. (2008) : Botanical insecticides, deterrents and repellents in modern agriculture and an increasingly regulated world. Ann. Rev. Entomol., 51: 45-66.

Ketoh, G.K.; Koumaglo H.K. and Glitho, I.A. (2005) : Inhibition of *Callosobruchus maculatus* (F.) (Coleopter: Bruchidae) development with essential oil extracted from *Cymbopogon schoenanthus* L. spreng (poaceae) and the Wasp *Dinarmus basalis* (Rondani) (Hymenoptera: Pteromalidae). J. Stored Prod. Res., 41: 363-371.

Komaitis, M.E.; Ifanti-Papatgragianni, N. and Melissari-Pangiotou, E. (1992) : Composition of the essential oil of marjoram (*Origanum majorana*, L.). Food Chemistry, 45(2): 117-118.

Lashgari, A.; Mashayekhi, S.; Javadzadeh, M. and Marzban, R. (2014) : Effect of *Mentha piperita* and *Cuminum cyminum* essential oil on *Tribolium castaneum* and *Sitophilus oryzae*. Archieves of Phytopathology and Plant Protection, 47: 3240329.

Lee, S.; Peterson C.J. and Coats J.R. (2003) : Fumigation toxicity of monoterpenoids to several stored product insects. J. Stored. Prod. Res., 39: 77-85.

Litchfield, J. and Wilcoxon, F. (1949) : A simplified method of evaluating dose-effect experiments. J. Exp. Ther., 96: 99-103.

Miyazawa, M.; Watanabe, H. and Kameoka, H. (1997): Inhibition of acetylcholinesterase activity by monoterpenoids with a *p*-methane skeleton. J. Agric. Food Chem., 45: 677-679.

Moino, A.J.r.; Alves, S.B. and Pereira, R.M. (1998) : Efficacy of *Beauveria bassiana* (Balsamo) Vuellemin isolates for untreated of stored grain pests. J. Appl. Ent., 122: 301-305.

Norambuena, C. G. S.; Urbina, A. I.; Figueroa, I. and Rodríguez, J.C. (2016) : Insecticidal activity of *Laureliopsis philippiana* (Looser) Schodde (Atherospermataceae) essential oil against *Sitophilus* spp. (Coleoptera Curculionidae). Chilean JAR Journal of Agricultural Research, 76(1): 330–336.

Nykanen, I. (1986) : High resolution gas chromatographic-mass spectrometric determination of the flavor composition of marjoram (*Origanum majorana* L.) cultivated in Finland. Z. Lebensm. Unters, Forsch., 183: 172-6.

Obeng-Ofori, D. and Reichmuth, C.H. (1999) : Plant oils as potentiation agents of monoterpens for protection of stored grains against damage by stored product beetle pests. Int. J. Pest Manag., 45(2): 155-159.

Okonkwo, E. U. and Ewete, F. K. (1999) : Toxicity of Dennettia tripetala and Piper guineense seed powders and oils against

Callosobruchus maculatus Fabricius Coleoptera: Bruchidae) and Sitophilus zeamais Journal of Herbs, Spices and Medicinal Plants, 1: 1-8.

Özcan, M.M. and Chalchat C.J. (2006) : Chemical composition and antifungal effect of anise (*Pimpinella anisum* L.) fruit oil at ripening stage. Ann. Microbiol., 56(4): 353-358.

Park, K.; Leeb, S.G.; Choib, D.H.; Park, J. and Young-Joon, A. (2003) : Insecticidal activity of constituents identified in the essential oil from leaves of *Chamaecyparis obtusa* against *Callosobruchus chinensis* (L.) and *Sitophilus oryzae* (L.). J. Stored Products Res., 39: 375-384.

Qi, Y.T. and Burkholder, W. (1981) : Protection of stored wheat from the granary weevil by vegetable oils. J. Econ. Ent., 74(5): 502-504.

Rozman, V.; Kalinovic, I. and Korunic, Z. (2007) : Toxicity of naturally occurring compounds of *Lamiaceae* and *Lauraceae* to three stored product insects. J. Stored Prod. Res., 43: 349-355.

Sarrou, E.; Chatzopoulou, P.; Dimassi-Theriou, K. and Therios, I. (2013) : Volatile constituents and antioxidant activity of peel, flowers and leaf oils of *Citrus aurantium* L. growing in Greece. Molecules, (18): 10639–10647.

Shaaya, E.; Kostjukovski, M.; Eilberg, J. and Sukprakam, C. (1997) : Plant oils as fumigants and contact insecticides for the control of stored product insects. J. Stored Product Res., 33: 7-15.

Sighamony, S.; Anees, I.; Chandrakala, T. S. and Osmani, Z. (1986) : Efficacy of certain indigenous plant products as grain protectants against *Sitophilus oryzae* (L.) and *Rhyzopertha dominica* (F.). J. stored Prod. Res., 22(1): 21-23.

Soliman, F.M.; Yousif, M.F.; Zaghloul, S.S. and Okba, M.M. (2009) : Seasonal Variation in the Essential Oil Composition of *Origanum majorana* L. Cultivated in Egypt. Z. Naturforsch, (64): 611-614.

SPSS for windows 7 (1995) : December 19, Network version, SPSS Inc., 1989-1995.

Srivastava, C. and Singh, D. (2002) : Study of phosphine resistance in *Rhyzopertha dominica* and *Callosobruchus maculates.* Indian J. Entomol.**, 64:** 377-378.

Subramanyam, B. and Hagstrum, D.W. (1995) : Resistance measurement and management. In Integrated Management of Insects in Stored Products (B. Subramanyam and D.W. Hagstrum, eds.). Marcel Dekker, New York, pp. 331-397.

Tapondjou, L.A.; Alder, C.; Bouda, H. and Fontem, D.A. (2002) : Efficacy of powder and essential oil from *Chenopodiini ambrosioides* leaves as post-harvest grain protectants against six stored product beetles. J. Stored Products Res., 38: 395-402.

Udo, I.O.; Epidi, T.t. and Osakwe, J.A. (2011) : Comparative efficacy of root bark and leaf powder of *Drocoenea arborea* for the control of two storage insect pests. Scientific Research and Essays, 6(7): 1473-1478.

Ullah, H.; Mahmood, A. and Honermeier, B. (2014) : Essential oil and composition of anise (*Pimpinella anisum* l.) with varying

seed rates and row spacing. Pak. J. Bot., 46(5): 1859-1864.

Wanida, A.; Angsumarn, C.; Anake, K. and Yooichi, K. (2012): Toxicity and repellency activities of the crude methanol effects of *Duabanga grandiflora* (Lythraceae) against *Sitophilus oryzae* (Coleoptera: Curculionidae). Pakistan J. Zol., 44(1): 227-232.

Zapata, N. and Smagghe, G. (2010) : Repellency and toxicity of essential oils from the leaves and bark of *Laurelia sempervirens* and *Drimys winteri* against *Tribolium castaneum*, Industrial Crops and Products, 3(32): 405-410.

Zarrad, K.; Chaieb, I.; Ben Hamouda, A.; Bouslama, T. and Laarif, A. (2017) : Chemical Composition and Insecticidal Effects of *Citrus aurantium* of Essential Oil and its Powdery Formulation against *Tuta absoluta* . Tunisian Journal of Plant Protection, (12): 83-94.

Ziga, L.; Matai, V. and Stanislav, T. (2012) : Efficacy of four essential oils against *Sitophilus granarius* (L.) adults after short-term exposure. African J. Agriculture Research, 7(21): 3175-3181.