Academic Journal of Entomology 13 (1): 08-15, 2020
ISSN 1995-8994

DOI: 10.5829/idosi.aje.2020.08.15

Evaluation of Some Essential Oils Combined with Ozone Gas as Potential Fumigant Against *Stegobium paniceum* (L.) (Coleoptera: Anobiidae) and Gibbium Psylloides (Czen.) (Coleoptera: Anobiidae)

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Abstract: Medicinal plants have devastating attacks of insect pests in stores. The objective of this research was to evaluate the effectiveness of clove buds, *Syzygium aromaticum,* patchouli, *Pogostemon cablin,* geranium, *Pelargonium graveolens* and lemon grass *Cymbopogon citrates* essential oils as fumigants against *Stegobium paniceum* and *Gibbium psylloides* adults. Furthermore, the toxicity of a combination of LC_{50} (111 and 204 ppm) ozone gas and LC_{50} essential oils on the adult insects was also investigated. Tested concentrations of Essential oils fumigation ranged from 20-90% with *G.psylloides* and 0.1-3% with *S.paniceum* for 24 h exposure period. Tested concentration of ozone ranged from 50-400 ppm for 30 min. to 4h exposure period. According to obtained results, mortality % increased with increasing of essential oil concentration. All tested oils at high concentrations (60% patchouli, 80% geranium and clove, 90% lemon grass for *G.psylloides*) and (3% for *S.paniceum*) were highly significant toxic ($p<0.05$) with both tested insects in which mortality ranged from 80-100%. Geranium (LC_{50}=0.34%) was the most effective oil fumigant against *S.paniceum* while patchouli (LC_{50}=37%) was the most effective oil fumigant against *G. psylloides*. Ozone at high concentrations (300 and 400 ppm) was highly significant toxic ($p<0.05$) with both tested insects in which mortality reached 100%. Applied combination between LC_{50} of geranium and patchouli essential oil with LC_{50} of ozone gas was additative and promising alternative against both tested insects in which give 100% mortality comparing with mortality % of applied LC_{50} of essential oil or ozone alone.

Key words: Essential oils・Ozone gas・Stegobium paniceum・Gibbium psylloides・Fumigation

INTRODUCTION

Botanicals; dried plants, roots, stems, leaves, seeds and flowers, have been used from the dawn of history as drugs or spices [1].

The drugstore beetle, *Stegobium paniceum* (L.) is a serious common pest of stored medicinal and aromatic plants which causes heavy losses in botanical warehouses [2].

Gibbium psylloides (Czen.), known as the hump beetle or the smooth spider beetle. They can cause fairly important damage in fabrics, books and stored food stuffs. Spider beetles can reduce the quality of commodities by contaminating them with webbing and droppings. The larvae bore into packaging and the grain itself, in addition to other materials such as grain sacks, leaving behind tell-tale holes [3].

The fumigant toxicity of essential oils owing to their high volatility attracted the attention of many researchers to replace conventional chemical pesticides in pest control. Many plant essential oils show a broad spectrum of activity against pest insects owing to their repellent, fumigant, insecticidal, antifeedent and oviposition deterrent. The fumigant toxicity of essential oils and their chemical constituents was first reported in 1960 [4].

In 1997, ozone was recognized as being generally safe (GRAS) for food contact applications in the United States [5, 6]. Since that time, interest in developing ozone applications in the food industry has increased, although some regulatory issues regarding ozone use for this purpose have not been resolved. Ozone (O_3) is a powerful oxidizing agent usually generated by electrical charges in air. It is generally accepted to be an unstable gas with a life span of about 20 min at room temperature; thus, it

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does not accumulate substantially without continual generation [7]. Electrical generation of ozone eliminates the handling, storage and disposal problems of conventionally used postharvest pesticides. An attractive aspect of ozone is that it decomposes rapidly to molecular oxygen without leaving a residue. These attributes make ozone an attractive candidate for controlling insects and fungi in stored products [8].

The objective of this research was to evaluate the effectiveness of essential oils as fumigants against *S.paniceum* and *G. psylloides*. Furthermore, studying the toxicity of a combination of ozone and essential oils to decrease exposure time of ozone without affecting its efficiency in killing insects.

MATERIALS AND METHODS

Experiments were performed in Plant Protection Research Institute- Agriculture Research Center- Egypt.

Insects: Adults of drugstore beetle, *Stegobium paniceum* (L.) (Coleoptera: Anobiidae) reared on coriander seeds and smooth spider beetle, Gibbium psylloidea (Czen.) (Coleoptera: Anobiidae) were reared on chamomile flowers kept in an incubator at 28±2°C and 65±5 % R.H.

Essential Oils: Essential oils of clove buds, *Syzygium aromaticum* (L.) (Myrtales:Myrtaceae). patchouli, *Pogostemon cablin* (Lamiales:Lamiaceae). Geranium, *Pelargonium graveolens* (Geraniales: Geraniaceae) and lemon grass *Cymbopogon citratus* (Poales:Poaceae) were purchased from Eden botanicals co., Petaluma, California, USA.

Essential Oils Fumigation Experiment: To investigate the fumigant toxicity of tested essential oils against adults of *S. paniceum* and G. *psylloides*, a laboratory experiment was carried out using a number of wooden fumigant chambers, each of approximately 6 L. Figure (1). A small electric device with 7 cm^2 disc was fixed in one of box sides. A glass slab was used to cover the box from the upper side. Each of the electric devices connected to electricity from outside source. Different concentrations (%) were prepared through dilution with petroleum ether solvent for each oil. Each concentration was replicated 3 times. Ten adults per replicate were put into 150 ml plastic jar that covered with fine small bores muslin to allow fumigant penetration. Then, the jars were introduced to the fumigant chambers. About 0.25 ml of each concentration was impregnated on to the disc. After evaporation of the solvent, the device was connected to electricity (220v) to help fumigation of oil. The duration of fumigation period used extend up to 24h. A control replicate with solvent only was used for comparing. Mortality percentage was then computed at the end of the experiment [9].

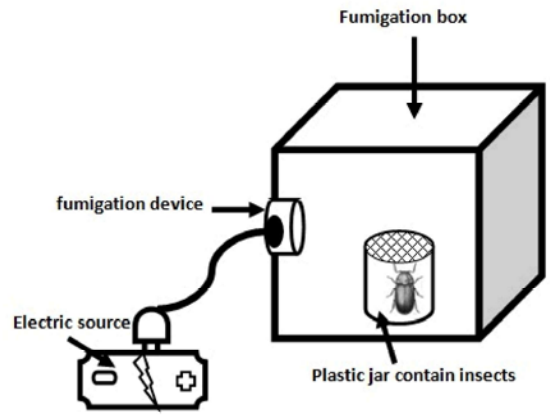

Fig. 1: Plastic jars covered with fine small bores muslin introduced into the fumigant chamber of approximately 6 L with fumigant electric device

Ozone Generator: Ozone production was generated via a controlled flow of oxygen through a corona discharge in the ozone generator (OZO Max Ltd, Shefford, Quebec, Canada) from purified extra dry oxygen feed gas at the laboratory of Food Toxicology & Contaminants, National Research Center. The ozone was fed into fumigation chamber where the ozone measurement and ozone treatment were conducted. Ozone was measured by an ozone monitor allows controlling the concentration in a selected range.

Treatment of Insects with Ozone: The treatment with ozone carried out inside gas-tight Dreshel flask of 2L capacity [10]. The flask was tightly plugged with 2 special glass stopper containing an inlet and outlet valve leading to vertical glass tube. The tube was long and worked as the gas inlet while the outlet closed with a plug. Ozone cylinder was applied for the ozone supply and connected to the inlet tube of the flask with a short hose. Valves were opened at the beginning of the treatment and left open until the desired gas concentration inside the flask was applied, as indicated by an oxygen analyzer (Servomex 570 A) connected to the outlet tube of the Dreshel flask by a short hose [11]. Ten adults of both insects were kept separately into fine porous bags (4×8 cm) and closed with rubber bands then introduced into Dreshel flask. Four replicates were used for each tested concentration of 100, 200, 300 and 400 ppm for

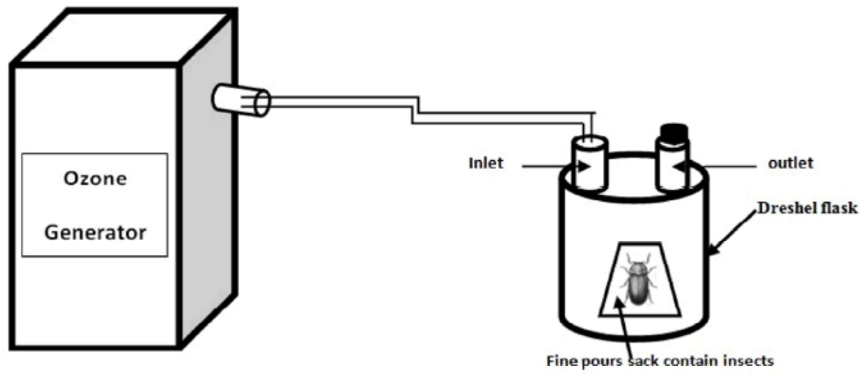

Fig. 2: Ozone generator connected to 2L Dreshel flasks with inlet and outlet valve contain fine porous sack with insects

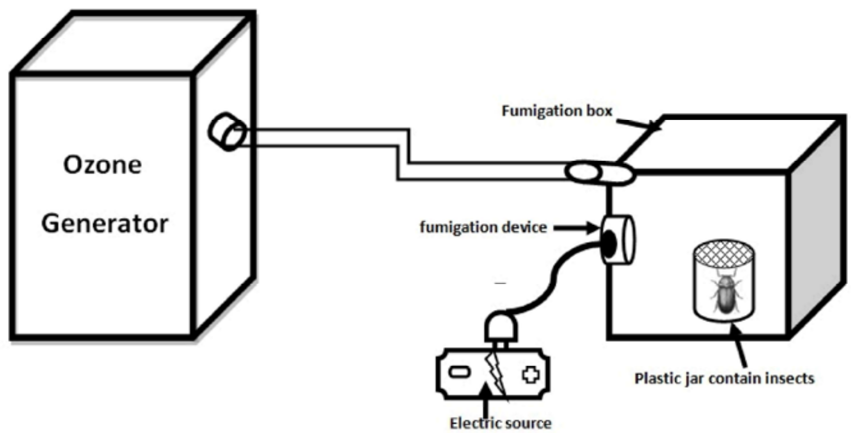

Fig. 3: Combined ozone gas - essential oil fumigation

5 different exposure periods (30 min. - 4h) Figure (2). Four untreated bags were kept as control for each insect. Mortality percentage was calculated after 24 h from the end of exposure period.

Fumigation of Insects with a Combination of Essential Oils and Ozone Gas: This experiment was conducted to evaluate LC_{50} of patchouli oil, (the most toxic against *G.psylloides*) and geranium oil (the most toxic against *S.paniceum*) combined with LC_{50} of ozone gas as potential alternative fumigant. Fifty adult from each tested insects were introduced separately into two plastic jars sealed with fine muslin then each jar was put inside a 15-liter test chamber (30*25*20) included a fixed small electric device with 7 cm^2 disc impregnated with LC_{50} of oil and connected to outside electric source and at the same time the chamber was connected to the ozone generator and ozone destruction unit by means of silicone tubing for 1h (the time which LC_{50} was calculated) Figure (3). Two fumigation boxes were used, one for each insect. Mortality percentage of both tested insects was calculated after exposure for LC_{50} of oil fumigant and ozone separately without combination for comparison. Mortality percentage for combination experiment was calculated after 24 hrs.

Statistical Analysis: Insect mortality data were subject to analysis of variance and differences using one way ANOVA test (a computer program costate). Mean values were adjusted by Duncan's Multiple Range test [12] at 0.05% level of significance with Statistical software version 6.3.0.3. The mortality pecentage was probit analyzed using a computer program named Ldp-line according to Finney [13]. From which the toxicity values (LC_{50}, LC_{90}) were estimated. Relative potency (RP), Resistance ratio (RR) and Slope values of tested compounds were also estimated and toxicity index.

RESULTS

Data Analysis: The probit statistics, estimate of LC_{50}, LC_{90} and the slope of regression lines of tested oils after 24 h and ozone gas after 1 h was represented in Tables (1-3). From probit analysis, it was found that patchouli oil was the most toxic oil against G. *psylloides* followed by geranium, clove then lemon grass respectively while the

Table 1: Toxicity values (%) of essential oils on *Gibbium psylloides*

Line name	LC_{50}	RR	R.P.%	Slope	LC_{90}
patchouli	37.138	1	100	7.4	55.4
geranium	43.6	1.2	85	5.2	76.8
Clove	48.839	1.3	76	3.5	114
lemongrass	66.922	1.8	55.5	7.3	100

Relative potency= LC_{50} of the most effective oil/ LC_{50} of the tested oil x 100

Relative potency (R.P.) compared with patchouli oil Resistance Ratio (RR) compared with patchouli oil

Table 2: Toxicity values (%) of tested oils on *Stegobium paniceum*

Line name	LC_{50}	RR	R.P	Slope	LC_{90}
geranium	0.34	1	100	1.2	3.8
patchouli	0.4	1.1	87.6	1.1	5.8
clove	0.63	1.8	54.9	1.4	4.9
lemongrass	0.63	1.8	54.7	1.1	8.3

Relative potency= LC_{50} of the most effective oil/ LC_{50} of the tested oil x100

Resistance Ratio (RR) compared with geranium oil

Table 3: Toxicity values (ppm) of ozone gas on *S. paniceum* and G. psylloides

Line name	LC_{50}	RR	R.P	Slope	LC_{90}
S. paniceum	111.7	1	100	1.99±0.37	488.5
G. psylloides	204.8	1.8	54.5	2.84±0.49	578.90

Relative potency= LC_{50} of the most effective pesticide/ LC_{50} of the tested pesticide x100

Resistance Ratio (RR) compared with *S. paniceum*

Table 4: The mortality percentage (mean ± SE) of tested oil fumigants against *Gibbium psylloides* after 24 hrs

Geranium		Patchouli		Clove		Lemon grass	
Conc. %	(%) mortality (mean ± SE)	Conc. %	(%) mortality (mean ± SE)	Conc. %	(%) mortality (mean ± SE)	Conc. %	(%) mortality (mean ± SE)
80	100±0^{a}	60	100±0^{a}	80	83.3±3.3^{a}	90	86.6±3.3^{a}
70	86.6±6.6^{a}	50	80±10^{b}	70	66.6±6.6^{b}	80	70±5.7^{b}
50	60±5.7^{b}	40	66.6±3.3^{b}	50	46.6±3.3^{c}	70	50±5.7^{c}
40	43.3±3.3^{c}	35	40±5.7^{c}	40	36.6±3.3^{cd}	60	36.6±3.3^{c}
30	20±5.7^{d}	30	23.3±3.3^{d}	30	26.6±3.3^{d}	50	20±5.7^{d}
20	10±5.7^{d}	20	16.6±3.3^{d}	20	13.3±3.3^{e}	40	6.6±3.3^{d}
LSD 0.5	15.7		16.2		12.5		14.5
Ctrl	0		0		0		0

Means followed by the different letters are significantly different from each other at $P<0.05$

most toxic oil against *S. paniceum* was geranium followed by patchouli, clove then lemon grass respectively. Ozone was more toxic against *S. paniceum* than *G. psylloides*. When probit regression lines of oils against tested insects were calculated, they showed a linear relationship between mortality percentage and concentration.

Essential Oil Fumigation: The mortality percentage of *G. psylloides* and *S. paniceum* adults after exposure to different concentration of four essential oil fumigants for 24 h are recorded in Tables (4&5) respectively. Mortality % significantly ($p<0.05$) increased with increasing of concentration. Geranium was the most effective oil fumigant against *S. paniceum* while with *G. psylloides* patchouli was the most effective oil fumigant.

All tested oils at high concentrations (60% patchouli, 80% geranium and clove, 90% lemon grass for *G. psylloides*) and (3% for *S. paniceum*) were highly significant toxic ($p<0.05$) with both tested insects in which recorded mortality ranged from 83.3-100% with *G. psylloides* and 80-100% with *S. paniceum*.

Ozone Treatment: The mortality percentage of *G. psylloides* and *S. paniceum* adults after exposure to different concentration of ozone gas for 5 different exposure periods (30 min. - 4h) are recorded in Tables (6&7) respectively. Mortality % increased with increasing of concentration and exposure time.

Table 5: The mortality percentage (mean ± SE) of tested oil fumigants against *Stegobium paniceum* after 24 hrs

Geranium		Patchouli		Clove		Lemon grass	
Conc. %	(%)mortality (mean ± SE)	Conc. %	(%)mortality (mean ± SE)	Conc. %	(%)mortality (mean ± SE)	Conc. %	(%)mortality (mean ± SE)
3	100±0[a]	3	83.3±6.6[a]	3	86.6±3.3[a]	3	80±5.7[a]
2	83.3±6.6[a]	2	76.6±8.8[ab]	2	70±5.7[b]	2	76.6±3.3[a]
1	70±5.7[ab]	1	66.6±6.6[ab]	1	63.3±3.3[b]	1	50±5.7[b]
0.5	56.6±3.3[b]	0.5	56.6±8.8[bc]	0.5	46.6±6.6[c]	0.5	43.3±8.8[b]
0.25	40±5.7[c]	0.25	43.3±6.6[cd]	0.25	26.6±3.3[d]	0.25	33.3±3.3[bc]
0.1	23.3±3.3[d]	0.1	23.3±3.3[d]	0.1	13.3±3.3[d]	0.1	20±5.7[c]
LSD 0.5	16.3	LSD 0.5	21.7	LSD 0.5	14	LSD 0.5	17.7
Ctrl	0		0		0		0

Means followed by the different letters are significantly different from each other at $P<0.05$

Table 6: The mortality percentage (mean ± SE) of Ozone gas against *Gibbium psylloides* after 24 h

	(%) mortality (mean ± SE)				
Conc.ppm	30 min	1 h	2 h	3 h	4 h
400	45±2.8[a]	82.5±4.8[a]	100±0[a]	100±0[a]	100±0[a]
300	25±2.8[b]	65±6.5[b]	77.5±2.5[b]	100±0[a]	100±0[a]
200	15±2.8[c]	47.5±6.3[c]	62.5±7.5[c]	82.5±4.8[b]	100±0[a]
100	0±0[d]	20±4[d]	25±2.8[d]	47.5±6.3[c]	95±2.9[b]
LSD 0.5	7.7	16.9	12.9	12.2	4.4
Ctrl	0	0	0	0	0

Means followed by the different letters are significantly different from each other at $P<0.05$

Table 7: The mortality percentage (mean ± SE) of Ozone gas against *Stegobium paniceum* after 24 h

	(%) mortality (mean ± SE)				
Conc.ppm	30 min	1 h	2 h	3 h	4 h
300	52.5±8.5[a]	85±2.8[a]	100±0[a]	100±0[a]	100±0[a]
200	40±4[ab]	62.5±8.5[b]	95±2.9[a]	97.5±2.5[a]	100±0[a]
100	30±4[b]	47.5±11[b]	77.5±6.3[b]	82.5±4.8[b]	100±0[a]
50	12.5±2.5[c]	25±2.8[c]	60±4[c]	70±4[c]	85±2.8[b]
LSD 0.5	16.4	22.5	12.4	10.4	4.4
Ctrl	0	0	0	0	0

Means followed by the different letters are significantly different from each other at $P<0.05$

Table 8: The mortality percentage of LC_{50} concentration ozone gas, essential oil fumigants alone and in combined with ozone gas against *Stegobium paniceum* and *Gibbium psylloides* adults after 24 h.

	Insects mean mortality %		
Treatment	*S. paniceum*	*G. psylloides*	Ctrl
Lc50 geranium essential oil	46.6	-	0
Lc50 patchouli essential oil	-	60	0
Lc50 ozone gas	46	52	0
Lc50 combined ozone geranium	100	-	0
Lc50 combined ozone patchouli	-	100	0

Ozone at high concentrations was highly significant toxic ($p<0.05$) with both insects in which mortality of *G. psylloides* ranged from 0-45%, 20-82.5%, 25-100%, 47.5-100%, 95-100% @ conc. 100,200,300&400 ppm for 30 min., 1h, 2h, 3h & 4h exposure period respectively. While mortality of *S. paniceum* ranged from 12.5-52.5%, 25-85%, 60-100%, 70-100%, 85-100% @ conc. 50,100,200,300 for 30 min., 1h, 2h,3h & 4h exposure period respectively.

Combined Ozone - Essential oil Fumigation: The mortality % of *S. paniceum* and *G. psylloides* fumigated with LC_{50} of geranium and patchouli essential oil respectively (the most toxic oil for each insect), LC_{50} of ozone gas alone and combined with LC_{50} of both oil separately was shown in Table (8).

Obtained results showed that applied combination between LC_{50} essential oil and LC_{50} ozone gas was additive and promising alternative against both tested insects in which give 100 % mortality comparing with mortality % of applied LC_{50} essential oil or ozone alone.

DISCUSSION

According to the obtained results from this research, both geranium and patchouli essential oils can provide a good alternative fumigant to control *S. paniceum* and *G. psylloides*. Previous studies tested the fumigant toxicity of plant oils against stored grain insects according to the traditional method described by Prates *et al.* [14] using filter paper such as a research performed by Ömer *et al.* [15] reported that the fumigant of *Mentha spicata* essential oil showed the highest toxicity with a mortality rate of 97.76% for S. paniceum.

However, in this research; the toxicity of plant oils was tested using a new fumigant technique. This technique was designed and used in a previous research by Sameeh *et al.* [9] in which fumigant toxicity of fixed and volatile oils of clove, cinnamon and moringa were tested against adults of *S. oryzae* and *T. castaneum* and the results showed that, this method was promising and give a good mortality % against both tested insects after 24 h from exposure also using the same technique Boraie [16] reported that clove and spearmint fixed oil exhibited strong fumigant toxicity against *S. paniceum* which cause 100% mortality at 10 % conc. after 24 h from exposure.

The mode of action of essential oils received attention from the research community. Enan [17, 18] have provided evidence that many essential oil constituents poison insects by blocking octopamine receptors. Octopamine synthesized from tyramine, is a neurotransmitter and neuromodulator in arthropods and may have neurohormonal influences as well.

The presence of volatile compounds having strong odor would have blocked the tracheal respiration of the insects leading to their death. Similar observation was made by Pugazhvendan *et al.* [19] the presence of volatile compounds is responsible for strong odour that could block the tracheal respiration of the insects leading to their death. The mode of action of oils was partially attributed to interference in normal respiration, resulting in suffocation [20].

Ozone at high concentrations was highly significant toxic ($p<0.05$) with both tested insects. Researchers have been recently focused on the application of O_3 as a fumigant to control stored-grain insects and microorganisms and to reduce myco-toxins. McDonough *et al.* [21] found that treatment of red flour beetle (*Tribolium castaneum*) and maize weevil (*Sitophilus zeamais*) by ozone at 1800 ppm for 120 min cause 100% mortality under laboratory conditions. Xinyi *et al.* [22] found that 0.42 g/m^3 ozone effectively killed adults of phosphine-susceptible and resistant strains of *T. castaneum*, *O. surinamensis*, *S. oryzae* and *S. zeamais*. In addition, phosphine-susceptible and-resistant strains of each species were equally susceptible to ozone. At this concentration, fumigation for 10 h can completely suppress adult progeny production of *Sitophilus* spp. and *O. surinamensis*, but not that of *T. castaneum* strains. Fuji *et al*. [23] reported that fumigation for 72 h with an average ozone generation rate of 100 g/h cause 100% for adults of *Cryptolestes ferrugineus*, *Tribolium castaneum*, *Sitophilus oryzae* and *Rhyzopertha dominca* held at the surface of the grain mass but not all of introduced adults were killed inside the grain mass. Ibrahim and Alahmadi [24] proved that 100 ppm ozone exposure for 1 hr cause 96.0 and 94.0 % mortality for B. incarnates larvae and adult respectively, 90.0 and 78.0 % mortality for larvae and adults of Tribolium spp respectively also treatment of wheat and cowpea with ozone resulted in slight missing of protein and lipid contents but increased sugar fractions of the tested materials.

There is a rare studies in controlling G. psylloides with fumigation as it considered often a minor insect and receives little attention as stored products pest but this insect becomes very important pests on chamomile [25]. But there is a study performed by Sayeda *et al.* [26] to evaluate the effectiveness of modified atmospheres (Mas) based on high CO_2 contents in controlling *G. psylloides*. Eggs, fourth instar larvae, pupae and adults, reported that the most effective MAs are those containing high level of gases such 40% and 80% (40% CO_2, 12 % O_2 and 48% N_2 and MA: 80% CO_2, 4%O_2 and 16% N_2) So, it is recommended to use these high levels of MAs in controlling all stages of this insect also Sayeda *et al.* [27] reported that Modified atmosphere (MA) containing 60% CO_2 is suitable for controlling adults and larvae of S. oryzae and T. castaneum, while MA containing 98% N_2 is suitable for controlling adults and larvae of T. castaneum in short time. The adults and larvae of S. oryzae were more sensitive to O_3 than those of T. castaneum. The exposure to 500 ppm O_3 for 7 days was preferable for controlling adults and larvae of S. oryzae and T. castaneum. MAs containing 60% CO_2 and 97% N_2 as well as 500 ppm O_3 had proven to be not effect in the milling and baking qualities of flour treated with it and N_2 reduce the quality of bread. The modified atmospheres and ozone are safe on mammals feed wheat grains exposed to these gases.

The obtained results showed that applied combination between LC_{50} essential oil and LC_{50} ozone gas was additive and promising alternative against both tested insects in which give 100 % mortality comparing with applied LC_{50} essential oil or ozone alone. Although the advantages of ozone as ecofriendly safe fumigant against stored grain insects that have a short half-life so reverts back to naturally occurring oxygen leaving no residue on the product and didn't affect chemical components of stored grain and kernel density that are vital for maintenance of milling and processing quality [28]. But its relatively rapid decay rate requires that it be continually generated lead to increase corrosion rates on metal components and degrade equipment such as rubber seals and electrical equipment at unacceptable rates [29, 30]. So there is urgent need to find potential safe alternative could combined with ozone to decrease exposure time without affecting its efficiency in killing insects.

The mode of action of ozone which is a very unstable molecule and rapidly decays into O_2 releasing a single oxygen atom that is highly reactive. This single oxygen reacts with the cell membrane of bacteria or virus attacking cellular components and disrupting normal cellular activity. (http://www.ozone-industries.co.uk/ozone_generation. html).

No previous data was recorded about studying the combination between essential oils and ozone fumigation against stored grain insects however combination between ozone and Co_2 to control stored grain insects was studied by Golam *et al*. [31] reported that the mixture of ozone with carbon dioxide can be as suitable fumigant for decreasing phosphine and methyl bromide under ambient storage conditions in penetration and empty-space fumigations.

CONCLUSION

Applied combination between LC_{50} essential oil and LC_{50} ozone gas was additive and promising alternative against *G. psylloides* and *S. paniceum* in which give 100 mortality % after 24 h comparing with applied LC_{50} essential oil or ozone alone.

ACKNOWLEDGMENTS

We thank the laboratory of Food Toxicology & Contaminants, National Research Center for technical support and providing the ozone generator.

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