Academic Journal of Entomology 16 (1): 01-10, 2023
ISSN 1995-8994

DOI: 10.5829/idosi.aje.2023.01.10

Evaluation the Efficiency of Two Forms of Plant Materials as Dustable Powder Formulation Against Red Flour Beetle *Tribolium castaneum* (Herbst) and its Effect on Storage of Wheat Flour

[1]Mai M.A. Gnedy and [2]Nilly A.H. Abdelfattah

[1]Central Agriculture Pesticides Laboratory, Agricultural Research Center, Dokki, Giza, Egypt
[2]Plant Protection Research Institute, Agricultural Research Center, Dokki, Giza 12619, Egypt

Abstract: The red flour beetle is one of the insects that are difficult to deal with and control due to its presence in wheat flour during storage. The research aims to find a safety and easy way to apply and deal with flour directly without compromising its properties and qualities. In this study were used botanicals materials (clove-oil and orange-oil, menthol-crystals and citric-acid-granules) to formulate a dustable powder. After passing all physical and chemical testing for dust powder, the formulations were biologically evaluated on three stages (larva, pupa and adult-stage) of red flour beetle *Tribolium castaneum* (Herbst). The results obtained indicated that, formulations of menthol-crystals and clove-oil exhibited insecticidal activity against adult, larvae and pupae stages of T. castaneum. The $Lc_{99\%}$ of adults' mortality was (22.7g/20g flour) for formulated clove and (3.29g/ 20g flour) for formulated menthol. The pupal stage was more tolerance than other stages. The residual effect and technological properties of flour are estimated for prepared menthol at a $Lc_{99\%}$ concentration.

Key words: Stored grains insects · Formulation · Insecticide · Residual effect · Technological properties

INTRODUCTION

The flour is subject to attack by several secondary insects as *Tribolium castaneum* (Herbst), *Tribolium confusum* (Jacquelin du Val), *Oryzaephilus surinamensis* (Linnaeus) and *Ephestia kuehniella* (Zeller) especially at the small scale farmer's level where storage conditions are usually inadequate to prevent or reduce insect attack. The flour insects cause severe quantitative and qualitative losses in developing countries, which have grave implications for the availability of foods, especially in those areas of the world where flour storage is poorly managed due to the lack of proper knowledge and technology.

Tribolium castaneum (Herbst), the red flour beetle, is a polyphagous, global pest found in flour mills and other locations where grain products and other dry foods are produced [1]. Both adults and larvae attack flours, cereals, nuts and dried fruits, especially because of their ability as cosmopolitan invaders of packaged food [2].

Insect pests significantly destroy stored grains of food in various parts of the world. Agriculturalists are looking for an alternative strategy that is effective, environmentally friendly and practically feasible, despite the fact that synthetic pesticides are effective and reliable. These factors include their high cost, non-biodegradability and negative effects on both people and the environment. Organic compounds in volatile plants could be an answer to problems with synthetic pesticides, such as those with resistance, availability, cost and health hazards, as well as deficiencies in hermetic storage, gamma irradiation and controlled environments [3].

Organophosphate and pyrethroid pesticides are mostly used to manage stored-grain insect pests [4]. In addition to pesticides, fumigants like methyl bromide, aluminium phosphide and magnesium phosphide are often employed to protect stored grains from insect pests [5]. The use of synthetic pesticides not only harms the environment, but also toxicity to human food systems [6]. The persistent use of chemical insecticides and fumigants

Corresponding Author: Nilly A.H. Abdelfattah, Plant Protection Research Institute, Agricultural Research Center, Dokki, Giza 12619, Egypt

has been linked to a number of issues, including resistance development, pesticide residues in food and harmful environmental consequences [7-10]. As a result, finding eco-friendly solutions to conserve grains is in great demand.

Due to the great performance of essential oils (EOs) in pest management, also their high efficacy, low toxicity, degradability, low residue, pest-sensitive and safety to humans, cattle, plants and natural enemies, (*Harmoniaaxyridis)* they have gained popularity in recent years [11, 12]. Essential oils as an eco-friendly possible alternative to chemical pesticides have been arousing our interest [13, 14]. The high insecticidal actions of EOs against a wide range of key agricultural pests have been linked to a number of components, most notably terpenoids and oxygenated terpenoids [15, 16].

In order to achieve a secure, simple and effective method of pest management that is also affordable, a pesticide must be transformed into a form that can be easily created, stored, transported and applied. This process is known as "pesticide formulation" to make it a product that can be stored, moved and used in an efficient, secure and cost-effective manner [17].

This work aims to find alternatives to pesticides in a form that can be easily and safely applied to mix with flour to protect it and control insects that attack it during storage.

MATERIALS AND METHODS

Plant Materials:
Menthol and citric-acid: Supplied by El-Goumhouria Company, Cairo, Egypt.
Clove and orange oils: Supplied by El-Gabry Pharmacy Essential Oils, Naser El–Dien St., Giza.

Solvents: Acetone and xylene were supplied by EL-Gomhoria Company, Cairo, Egypt.

Diluent: Were purchased from local market

The Physico-Chemical Properties of Active Ingredient:
Solubility: At 20°C, the total solubility or miscibility of one gram of active ingredient was tested by measuring the volume of distilled water, acetone and xylene [18]. The following equation was used to compute the percent solubility:

% solubility = W/V x 100

where; W= active ingredient weight, V= volume of solvent required for complete solubility].

Free Acidity or Alkalinity: It was determined using the same methodology set in FAO and WHO guideline MT 191 [19].

The Physico-Chemical Properties of Diluent:
Dry sieve test: It was determined according to (CIPAC, MT 59.1) [20].

Free Acidity or Alkalinity: It was determined using the same methodology set in FAO and WHO guideline MT 191 [19].

Bulk Density: Was acquired through FAO and WHO guideline MT 186 [21].

Preparation of Botanical as Dustable Powder: The botanical active ingredients were created into dustable powder formulations by the dry mix method, which involves mixing the active ingredient with the best diluent available [22]. Because menthol and citric acid have 50% (wt/wt) active components and clove oil and orange include 20% (wt/wt) oil, the mixture was thoroughly mixed before passing through a 74 micron screen. The manufactured dusts were subjected to the following tests:

Physico–Chemical Properties of Dustable Powder Formulations:
Dry sieve test: as before determined.
Free acidity or alkalinity: as before determined.
Bulk density: as before determined.
Bioassay:

Insect Cultures: The stocks of insects used in this experiments; the red flour beetle, Tribolium castaneum (Herbst) were collected from stock cultures maintained at the cereals and stored product pests Department, Plant Protection Research Institute, whereas they were reared at 28±2°C and 65±5% R.H. on whole wheat flour for at least two months.

Experimental Procedures of Bioassay: Twenty grams of wheat flour were put in small plastic cup (120 ml). Mixed well different weights of each formulation (0.5, 0.6, 0.7 and 0.8 g) for plant wheat flour, 20 unsexed adult of *T. castaneum* and 20 larvae were infested then a cup was tightly closed and putted in incubator at 29.4±0.5°C and

65±5% R.H. Mortality percentage was recorded after 7 days later as an initial experiment. The main experiment was repeated in the same procedure of initial experiment for 10 days at 32±2°C and 65±5% R.H. with dustable powder (0.8, 1.0, 1.2 and 1.4 g) for plant materials per 20 g of wheat flour for 20 unsexed (two weeks old) adults and 20 (15-days-old) larvae but 20 (0 – 1-day old) pupae were treated with (0.2, 0.3 and 0.4 g) for the same plant materials per 20 g of wheat flour for 3 days at the same previous conditions. Each stage was treated separately and each weight was replicated three times and one group was not treated as a control group. Mortality percent and number of F_1 of progeny and its reduction were recorded.

Residual Effect: The determination of the residual effect of the prepared dustable powder and the raw (pure) material on 20 unsexed (two weeks old) adults stage of red flour beetles after 10 days was carried out after wheat flour stored for zero, one, two and three months of treatments at 32±2°C and 65±5% R.H by the same procedure of main experiment and in each month's treatment, the test was repeated three times with the same wheat flour mixed with menthol according [23]. Each treatment was 3 replicates other than control group.

Technology Properties Test: The effect of mixing behaviors of menthol 50 % dustable powder with $L_{C99\%}$ concentration on technological properties of the flour at zero month (immediately after the treatment), A Mixolab analyzer (Chopin, Villeneuve-la-Garenne, France) was used to perform Mixolab assessments for flour according to ICC [24]. A mixing bowl of 300 g and 80 RPM mixing speed were used. The Mixolab device was used to determine the water absorption (g/kg), dough stability time (min), dough development time (min).

Data Analysis: "Ehabsoft" was used for the statistical analysis of bioassay [25]. Data analysis obtained of residual effect was analyzed as one/two way ANOVA, using Proc ANOVA in SAS (Anonymous) [26] and means were compared by LSD (P = 0.05 level) in the same program.

RESULTS AND DISCUSSION

The Physico-Chemical Characteristics of Compounds as Active Ingredient (Formulation Part): The data in Table (1) indicated the physical properties of compounds except for citric acid they were all insoluble in water. In addition to Citric acid and menthol were insoluble in xylene according to Gnedy [27]. Whereas, clove oil and orange oil were medium solubility in xylene and all compounds except citric acid were completely soluble in acetone. On the other hand, all compounds had a low free acidity, but citric acid had a high free acidity as percentage sulfuric acid (45.6), therefore, other additive materials necessary for preparation of suitable formulations, such as diluents, surfactants should be acidic to ensure optimal compatibility and prevent any chemical interactions between the formula's components, according to Gnedy [27].

To choose a suitable diluent, the pesticide formulator should be aware of the diluent's qualities that correspond to the active ingredient's properties. Furthermore, the physico-chemical characteristics of the diluent utilised are shown in Table (2). It passed successfully through 74 micron test sieve according to (CIPAC, MT59.1) [20]. The diluent also showed it was slightly acidic the % free acidity was (0.249), therefore it was suitable as a diluent for botanical active ingredient which showed acidic values. In addition to, the diluent's bulk density before and after compaction was 0.5 and 0.91 respectively, which is in accordance with the WHO guideline [28], which states that the percent fluctuation in bulk density between before and after compaction should not exceed 60 %. According to previous data in Table (2) wheat flour was suitable as diluent for preparing dustable powder. Accordingly Batta [29] prepared the formulation of *Metarhizium anisopliae* conidia contained fungal conidia and a dust carrier of wheat flour at 1:4 ratio (W/W) for evaluation of *Sitophyllus oryzae* adult mortality.

Also, data presented in Table (2) indicate that all tested dustable powder passed successfully through 74 micron test sieve, which complies with FAO, WHO [30] recommendations. The bulk density acquired before and after compacting (packed) was compatible with the WHO standard [28] which indicated that the bulk density of the powder after compaction should not be greater than 60 % of the value obtained before compaction. Furthermore, except for citric acid, all developed formulations as dustable powder had modest acidic properties derived from free acidity as sulfuric acid.

Biological Activity as Insecticide: Data in Table (3) indicated the initial effect of treatment with the two different formulation of dustable powder made from two forms of plant material; oil; 20% (w/w) and crystal or granule; 50% (w/w). For concentrations of 0.5 to 0.8 g/20 g flour, formulated orange, clove, citrate and menthol were mixed with flour for 7 days (in 120 ml plastic cup)

Table 1: Physico-chemical properties of the active ingredients

Plant ingredients	Solubility % (wt./v.) Water	Xylene	Acetone	Free Acidity % As H_2SO_4
Citric acid granule	100 soluble	Insoluble	10	45.6
Menthol crystal	Insoluble	Insoluble	50	0.049
Clove oil	Insoluble	50	100	0.196
Orange oil	Insoluble	50	100	0.49

Table 2: Physical properties of the chosen diluent and the prepared dustable powder formulations

Compound	Free acidity % as H_2SO_4	Dry sieve test through 74 micron	Bulk density Before compacting	After compacting
Wheat flour (diluent)	0.249	Passed	0.5	0.91
Citric acid	46.77	Passed	0.53	0.67
Menthol	0.098	Passed	0.38	0.67
Clove	0.392	Passed	0.5	0.83
Orange	0.294	Passed	0.42	0.91

Table 3: Preliminary experiment of formulated tested materials on adults and larvae stages of *T. castaneum* after 7 days at 29.5±0.5° C and 65±5% R.H

Stage	Adults			Larvae
Conc. 0.5-0.8 g/20 g flour	Mortality (%)	No. of (F_1) progeny	Reduction (%)	Mortality (%)
Orange	5.0-13.13	32.33-64.33	67.00-83.42	1.67-8.33
Citrate	3.0-13.13	58.33-119	38.97-70.08	1.67-5.0
Clove	1.67-3.33	10-12	91-92.42	6.67-11.67
Menthol	5.0-27.5	0-0	100-100	7.5-22.5
Control	0	195	-	0

and infested with *T. castaneum* adults and larvae then tightly closed. The results showed that formulation of clove 20 % and menthol 50 % dustable powder had the highest toxic effect, with 91 to 92.42 percent reductions in F_1 progeny in adults and higher larval mortality percentages than formulation of orange 20 % and citrate 50 % dustable powder. As general results of Tables (3), it could be said that prepared clove and menthol were more active than prepared orange and citrate.

The higher temperature, concentration and duration were conducted than in the preliminary experiment to give a highly responsive effect of the used materials. Different weights (0.8, 1.0, 1.2 and 1.4 g/20 g flour) putted in a 120 ml volume closed cup at 32±2°C for 10 days. When formulated clove material was used at 32±2°C for 10 days, the mortality rate reached 72.5% at 1.4 g for adults and 100% reduction in F_1 progeny, also, 52.5% at 1.4 g for larvae and 75% reduction in F_1 progeny (Table 4). While adult mortality rates reached 80.33% at 1.4 g and larval mortality rates reached 52.5% at 1.4 g, there was a 100% reduction in F_1 progeny in both stages when formulated menthol was used (Table 5). So, formulated clove and menthol can be used as a green insecticide for control of the flour beetle. EOs are acknowledged by the American FDA (Food and Drug Administration) as being less dangerous than synthetic pesticides [31]. Numerous studies have examined the impact of EOs on growth inhibition, insecticidal, ovicidal and repellant properties [31-34]. Methods for measuring insecticidal activity include: a) fumigation, in which EOs can be consumed, breathed, or absorbed; b) contact, in which EOs should penetrate the insects' cuticles; and c) ingestion [35, 36]. It has been also plant metabolites' method of action can prevent acetylcholinesterase (AChE) from hydrolyzing acetylcholine, a neurotransmitter essential for the central nervous system's signalling [37].

Data obtained in Tables (4 and 5) also showed that pupae had a higher response than adult and larvae stages where the concentrations used were lower (0.2, 0.3 and 0.4 g/20 g flour) for 3 days in a cup at 32±2°C. From the above results, we found that formulated menthol was more active and more toxic than formulated clove in controlling larvae, pupae and adults of *T.* castaneum insects.

Regnault-Roger *et al.* [38]; De Oliveira *et al.* [39] and Singh *et al.* [3] recorded that carvacrol, linalool, alpha-pinene, menthol, cinnamaldehyde, eugenol, 1-8 cineole, geraniol and limonene are some of the components of EOs that have demonstrated insecticidal action against various pests.

Table 4: Response of adults, larvae and pupae stages of *T. castaneum* of clove 20% dustable powder formulation at 32±2°C and 65±5% R.H

Stage	Conc. g/20g flour	Mortality %	No. of (F_1) progeny	Reduction %	$Lc_{50\%}$	$Lc_{99\%}$	Lower	Upper	X^2	Slope	H
Adults	0.8	60±7.64	9.33±1.76	92.93	0.58	22.7	-	-	0.77	1.46±0.71	0.38
	1.0	60±10.41	1±1	99.24							
	1.2	67.5±10.10	0±0	100							
	1.4	72.5±7.21	0±0	100							
	Control	20±0	132±0	-							
Larvae	0.8	17.5±4.33	11.67±0.67	41.67	1.33	4.95	3.31	11.6	0.16	4.08±0.75	0.08
	1.0	31.67±4.41	9±0.58	55							
	1.2	43.33±3.33	5±0	75							
	1.4	52.5±1.44	5±0.58	75							
	Control	0±0	20±0	-							
Pupae	0.2	0±0	20±0	0	-	-	-	-	-	-	-
	0.3	0±0	20±0	0							
	0.4	45±0	11±0	26.67							
	Control	25±0	15±0	-							

*Lc=Lethal Concentration

Table 5: Response of adults, larvae and pupae stages of *T. castaneum* of menthol 50% dustable powder formulation at 32±2°C and 65±5% R.H

Stage	Conc. g/20g flour	Mortality %	No. of (F_1) progeny	Reduction %	$Lc_{50\%}$	$Lc_{99\%}$	Lower	Upper	X^2	Slope	H
Adults	0.8	58.33±4.41	0±0	100	0.63	3.29	2.26	9.12	4	3.25±0.77	2
	1.0	80±5	0±0	100							
	1.2	80.33±3.33	0±0	100							
	1.4	80.33±1.67	0±0	100							
	Control	20±0	132±0	-							
Larvae	0.8	31.67±3.33	4.33±1.20	78.33±6.01	1.24	14.2	5.23	1120	0.84	2.19±0.70	0.42
	1.0	45±2.88	2.5±0.29	87.5±1.44							
	1.2	50±8.66	2±1.15	90±5.77							
	1.4	52.5±7.21	0±0	100							
	Control	0±0	20±0	-							
Pupae	0.2	40±2.88	12±0.58	20±3.85	0.23	0.72	0.62	0.91	5.96	4.65±0.47	2.98
	0.3	65±0	7±0	53.33±0							
	0.4	95±0	1±0	93.33±0							
	Control	25±0	15±0	-							

*Lc=Lethal Concentration

Lethal and sub lethal concentration values and parameters of the toxicity ldp line for the adults, larvae and pupae of *T. castaneum* exposed to clove and menthol has been formulated were presented in Tables (4 and 5). The results showed that the lethal concentration of formulated clove required to achieve 99 percent mortality for the adults of *T. castaneum* was 22.7 g/20 g flour and 4.95 g/20 g flour for larvae. Also, the results showed that the lethal concentration of formulated menthol required to achieve 99 percent mortality for the adults of *T. castaneum* was 3.29 g/20 g flour and 14.24 g/20 g flour for larvae, while for pupae it was 0.73 g/20 g flour when treated with formulated menthol. So from these results, the efficacy of formulations of menthol and clove on different stages of T. castaneum insect depending on their Ldp line after 10 days was according $Lc_{99\%}$ values for adult insects. For both prepared materials, the slope values were higher in the formulation of menthol than in the formulation of clove, indicating that the toxicity line is sharper in the menthol than in the clove one. On the other hand, could say that the larvae were more resistant to menthol, followed by the adults, but the pupae were more responsive.

The results in Table (6) contain the results of the raw menthol to compare them with the results of menthol after it has been formulated as a dustable powder. It is logical to find that the results of the raw material are higher and more efficient than the formulated material, knowing that the results of the dustable powder material in the case of the adults insect and pupae did not decrease much from the raw material, but rather gave completely satisfactory results. On the other hand, we noticed that the raw material (before formulation) has difficult properties in dealing with flour, as its granules of crystal gather and gather flour around them, which makes the material difficult to disintegrate to deal with,

Table 6: Response of adults, larvae and pupae stages of *T. castaneum* of the raw menthol at 32±2°C and 65±5% R.H

Stage	Conc.g/20 g flour	Mortality %	No. of (F_1) progeny	Reduction %	$Lc_{50\%}$	$Lc_{99\%}$	Lower	Upper	X^2	Slope	H
Adults	0.8	55±5	0±0	100	0.74	1.63	1.45	1.98	3.04	6.87±0.95	1.52
	1.0	85±8.66	0±0	100							
	1.2	93.33±6.67	0±0	100							
	1.4	95±5	0±0	100							
	Control	0±0	68.33±4.4	-							
Larvae	0.8	68.33±16.91	3±0.81	84.21	0.60	2.06	1.67	3.25	2.45	4.37±0.87	1.22
	1.0	87.5±4.33	1.67±1.20	91.22							
	1.2	87.5±7.21	1±0	94.73							
	1.4	95±0	1±0	94.73							
	Control	1.67±1.67	19±0	-							
Pupae	0.2	77.5±7.21	4.5±1.44	52.5	0.17	0.28	-	-	5.33	10.04±2.64	5.33
	0.3	100±0	0±0	75							
	0.4	100±0	0±0	75							
	Control	20±0	16±0	-							

*Lc=Lethal Concentration

unlike powder, which is easy and smooth to deal with even after adding it to flour and this is one of the advantages of converting the material from raw to powder. This is in addition to the economic value that comes from reducing the use of raw materials directly.

To determine its ability to protect wheat flour from *T. castaneum* for long periods after (zero, one, two and three months), the residual effect test was performed using concentration-caused mortality with 99% against adults insect. Over the course of a month, the test was repeated three times in flour mixed with dust powder. The residual effect of the substance is unaffected by the length of time or the number of times it is used; rather, the effect is continuous as long as the containers have tight seals (Figures 1 and 2), but the fluctuation in the results is caused by fluctuated the temperature between ± 2°C. Although essential oil-based pesticides offer a number of benefits and have been shown to be effective against particular insect species, they are very volatile in certain temperature and pressure conditions. By encapsulating those using existing techniques, their shortcomings, which are caused by their high volatility and rate of deterioration, can be reduced [40].

The statistical analysis showed that there was a significant difference between the raw (pure) material and dust powder, but there was no significant difference between months or between times Table (7). This indicates that the both materials can be used several times in controlling the insect and protecting the flour. The results ranged between 68.33 and 100 percent mortality rates of insects during 3 months of storage and 3 uses of the same packages that were previously used for each month separately compared with control recorded 0 percent mortality.

Technological Properties of Flour: Data in Table (8) shows the results of some technological tests on prepared dustable powder of menthol-treated flour and untreated flour at zero month (immediately after treatment). A comparison of the results of treated and untreated flour revealed that the arrival time, dough development and extensibility remained unchanged, indicating that dustable menthol powder had no effect on flour. On the other hand, water absorption percentage, stability time and elasticity were lower in treated flour than untreated flour, this characters also different between the types of flour in general and it is possible that the results of the different properties of the treated sample will converge with the results of the control sample if we leave the flour packages open for a while and thus any negative effect may be vanished. On the other hand a result of the natural properties of the material, it is expected that the raw material will be more difficult to work with in terms of flexibility, softness, stability, elasticity and water absorption than the prepared material. In the same trend in some properties of treated flour with dill oil Zinhoum [41] concluded that the effect of the dill oil treatment on water absorption (%), mixing time (min) and dough stability (min) was reduced, as was the dough's extensibility under dill oil compared to the control. On the other hand, the stability of wheat flour was increased to 18 minutes in dill oil as compared to the control value of 5 minutes.

In this context and from the results of this work, it can be said that the can be prepare formulation from different form of plant materials and can be applied with different ways. Dustable powder menthol can be considered a green insecticide and that it is safe to mix with flour, as it is naturally used in food and drink, where it the active

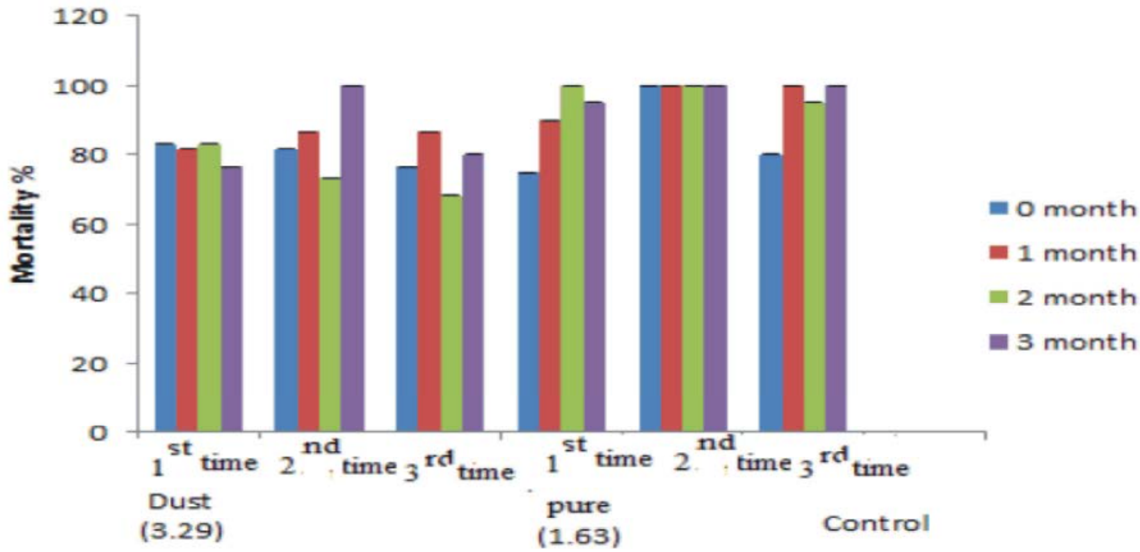

Fig. 1: Effect of three times on residual effect of $Lc_{99\%}$ of menthol on wheat flour at 32±2°C and 65±5% R.H

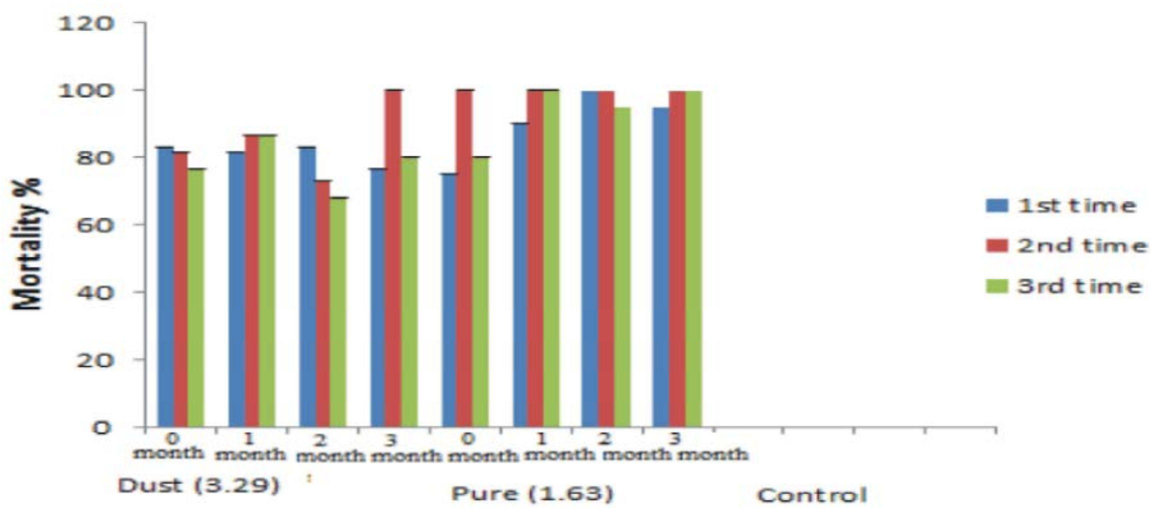

Fig. 2: Effect of months on residual effect of $Lc_{99\%}$ of menthol on wheat flour at 32±2°C and 65±5% R.H

Table 7: The statistical analysis of different variances of residual effect experiment with $Lc_{99\%}$ of menthol on wheat flour at 32±2°C and 65±5% R.H

Variance	Tukay arrangement				F value	L.S.D
Treatment	Dust[b]		Pure[a]	Control[c]	780.76	5.314
Months	Zero[a]	One[a]	Two[a]	Three[a]	1.74	6.1361
Times	1st time [a]		2nd time [a]	3rd time [a]	2.15	5.314

L.S.D.= Least Significant Difference

Table 8: Technological properties of untreated and treated flour mixed with $Lc_{99\%}$ of menthol 50% dustable powder formulation immediately after treatment

Parameter	Water absorption %	Arrival time (min)	Dough development (min)	Stability time (min)	Degree of softening (B.U.)	Elasticity (B.U.)	Extensibility (mm)
Treated	53.0	1.0	1.5	2.0	50	440	135
Control	63.0	1.0	1.5	3.0	70	710	135

ingredient in mint, in addition to its low cost. On the other hand, it was found that it can be used several times to preserve flour and to control insects, if any. Also find that the formulation of the material into other forms gives it better properties than the raw material in dealing with baking, the technological properties of flour and the ease of its application to preserve the flour from insect infestation during storage. Some research supported our work and said; plant-derived botanicals hold promise as an alternative to synthetic insecticides to lessen the negative impact of the pesticide on the environment. It is well known that volatile organic molecules in plants have insecticidal effects. Since plant volatile organic compounds are affordable, easily accessible to farmers, eco-friendly and have low mammalian toxicity, they may be suggested as a viable alternative to synthetic pesticides [3]. Pasha [42] observed that camphor, clove and mint displayed the greatest repellent behavior for the confused beetle, T. confusum. As for the mode of toxicity of the insect, it can be said that toxicity, repellency, antifeedant or feeding deterrent, fumigant, growth inhibitor, inhibition of reproductive behavior and loss of fecundity and fertility are the ways phytochemicals affect insects [43, 44].

CONCLUSION

Menthol (crystal) and clove (oil) were formulated as dustable powder. These formulations passed successfully all physico-chemical properties of dustable powder and then tested against *T. castaneum* insect. Formulation of menthol and clove can be used as insecticide for the cereals and stored product pests. Formulated menthol was highly active in toxicity than formulated clove. Pupae stage was more susceptible than larvae and adults. The residual effect and technological properties of flour after treatment with formulated menthol gave positive results. Menthol can be added to flour for protection and control of larvae, pupae and adults stages of red flour beetle, *T. castaneum*.

REFERENCES

1. Hussain, H.B.H. and N.A.H. Abdelfattah, 2013. Weight loss of wheat flour caused by *Tribolium castaneum* (herbst) and *Oryzaephilus surinamensis* (L.) infestation under laboratory conditions. Bulletin of Entomology Society of Egypt, 90: 183-194.
2. Mowery, S.V., M.A. Mullen, J.F. Campbell and A.B. Broce, 2002. Mechanisms underlying saw-toothed grain beetle *Oryzaephilus surinamensis* (Coleoptera : Silvanidae) infestation of consumer food packing materials. Journal of Economic Entomology, 95: 1333-1336.
3. Singh, K.D., J.M. Adesina, B. Rupjyoti, S. Dinabandhu and R. Yallappa, 2021. Main plant volatiles as stored grain pest management approach: A review, Journal of Agriculture and Food Research, 4: 100127.
4. Sousa A.H., L.R.D.A. Faroni, R.N.C. Guedes, M.R. To´ tola and W.I. Urruchi, 2008. Ozone as a management alternative against phosphine-resistant insect pests of stored products. Journal of Stored Product Research, 44: 379-385.
5. Olivero-Verbel, J., L.S. Nerio and E.E. Stashenko, 2010. Bioactivity Against *Tribolium castaneum* Herbst (Coleoptera: Tenebrionidae) of *Cymbopogon citratus* and *Eucalyptus citriodora* essential oils grown in Colombia. Pest Management Science: formerly Pesticide Science, 66: 664-668.
6. Caballero-Gallardo, K., J. Olivero-Verbel and E.E. Stashenko, 2011. Repellent activity of essential oils and some of their individual constituents against *Tribolium castaneum* Herbst. Journal of Agriculture and Food Chemistry, 59: 1690-1696.
7. Benhalima, H., M.Q. Chaudhry, K.A. Mills and N.R. Price, 2004. Phosphine resistance in stored-product insects collected from various grain storage facilities in Morocco. Journal of Stored Product Research, 40: 241-249. https://doi.org/10.1016/S0022-474X(03)00012-2
8. Kljajiae, P. and I. Periae, 2009. Residual effects of deltamethrin and malathion on different populations of *Sitophilus granarius*(L.) on treated wheat grains. Journal of Stored Product Research, 45: 45-48. https://doi.org/10.1016/j.jspr.2008.07.004
9. Attia, M.A., T.F. Wahba, M.I. Mackled and M.S. Shawir, 2017. Resistance Status and Associated Resistance Mechanisms to Certain Insecticides in Rice Weevil *Sitophilus oryzae*(Coleoptera: Curculionidae). Alexandria Journal of Agriculatural Sciences, 62: 331-340.
10. Bomzan, D.P., M.L. Bhavya, A.G.S. Chandu, S. Manivannan, G. Lavanya, K. Ramasamy and A.A. Pasha, 2018. Potential of pyrethroid-synergised pyrethrum on stored product insects and implications for use as prophylactic sprays. Journal of Food Science and Technology, 55: 2270-2278. https://doi.org/10.1007/s13197-018-3144-8

11. Pavela, R., 2018. Essential oils from *Foeniculum vulgare* Miller as a safe environmental insecticide against the aphid *Myzus persicae* Sulzer. Environmental Science and Pollution Research, 25: 18708-18716.https://doi.org/10.1007/s11356-018-1398-3
12. Benelli, G., R. Pavela, R. Petrelli, L. Cappellacci, F. Bartolucci, A. Canale and F. Maggi, 2019. *Origanumsyria cum*subsp. *Syriacum*: from an ingredient of Lebanese 'manoushe' to a source of effective and ecofriendly botanical insecticides. Industry Crop Product, 134: 26-32. https://doi.org/10.1016/j.indcrop.2019.03.055
13. Pavela, R. and G. Benelli, 2016. Essential oils as ecofriendly biopesticides ?Challenges and constraints. Trends Plant Science, 21: 1000-1007. https://doi.org/10.1016/j. tplants.2016.10.005
14. Pavela, R., G. Benelli, L. Pavoni, G. Bonacucina, M. Cespi, K. Cianfaglione, I. Bajalan, M.R. Morshedloo, G. Lupidi, D. Romano, A. Canale and F. Maggi, 2019. Micro emulsions for delivery of Apiaceae essential oils towards highly effective and ecofriendly mosquito larvicides? Industrial Crops and Products, 129: 631-640. https://doi.org/ 10.1016/j.indcrop.2018.11.07
15. Xie, Y., K. Wang, Q. Huang and C. Lei, 2014. Evaluation toxicity of monoterpenes to subterranean termite, *Reticuliterme schinensis* Snyder. Industrial Crops and Products, 53: 163-166. https://doi.org/10.1016/j.indcrop.2013.12.021
16. Zhang, Z., T. Yang, Y. Zhang, L. Wang and Y. Xie, 2016. Fumigant toxicity of monoterpenes against fruitfly, *Drosophila melanogaster*. Industrial Crops and Products, 81: 147-151. https://doi.org/10.1016/j.indcrop.2015.11.076
17. Hazra, D.K. and K. Purkait, 2019. Role of pesticide formu-lations for sustainable crop protection and environment management: A review. Journal of Pharmacognosy and Phytochemistry, 8(2): 686-693. http://krishikosh.egra nth.ac.in/handle/1/5810096400
18. Nelson, F.C. and G.W. Fiero, 1954. A selected Aromatic Fraction Naturally Occurring in Petroleum as Pesticides Solvents. Journal of Agriculture and Food Chemistry, 14(2): 1765-1737.
19. Food and Agriculture Organization, World Health Organization 2002. Manual on development and use of pesticides, prepared by FAO/WHO joint meeting on pesticide specification (JMPS), 1st Ed., Rom.
20. Collaborative International Pesticides Analytical Council Limited, 2002. CIPAC handbook. Physic-chemical methods for technical and formulated pesticides, 2002.
21. Food and Agriculture Organization and World Health Organization, FAO and WHO, 1st 2nd rev. MT 191, 2010. Manual on Development and Use of FAO and WHO Specifications for Pesticides.
22. Furmidge, C.G.L., 1972. General principles governing the behavior of granular formulation, Pesticide Science., 3(6): 745-751. https://doi.org/10.1002/ps.2780030612
23. Abdelfattah, N.A.H. and M.A. Gnedy, 2021. Preparation of some safe synthetic botanical materials as fumigant tablets as alternatives for hazard phostoxin for controlling cowpea beetle *Callosobruchus maculatus* (Coleoptera: Chrysomelidae) infesting leguminaceous seeds. Egyptian Journal of Plant Protection Research Institute, 4(4): 545-553.
24. ICC, 2010."Standard Methods of the International Association for Cereal Chemistry 173". International Association for Cereal Science and Technology, Vienna.
25. Bakr, E., 2000. LdP line software. http://www.ehabsoft.com/ ldpline.
26. Anonymous, SAS., 2003. Statistics and graphics guide, release 9.1. SAS Institute, Cary, North Carolina, 27513, USA.
27. Gnedy, M.M.A., 2016. Formulation of synthetic botanical bioactive compounds and determination of their pesticidal effectiveness. Ph.D. Thesis, Faculity of Agriculture, Fayoum University.
28. World Health Organization, 1979. Specification of Pesticides Used in Public Health, 5th Ed. Geneva.
29. Batta, Y.A., 2004. Control of rice weevil (*Sitophilusoryzae*L., Coleoptera: Curculionidae) with various formulations of M*etarhizium anisopliae*, Crop Protection, 23: 103-108.
30. Food and Agriculture Organization and World Health Organization, 2010. FAO and WHO, 1st 2nd rev. MT 186, Manual on Development and Use of FAO and WHO Specifications for Pesticide.
31. Hikal, W.M., R.S. Baeshen and H.A.H.S. Ahl, 2017. A. Botanical insecticide as simple extractives for pest control. Cogent Biology, 3: 1404274. doi: 10.1080/23312025.2017.1404274
32. Ikbal, C. and R. Pavela, 2019. Essential oils as active ingredients of botanical insecticides against aphids. Journal of Pest Science, 92: 971-986. doi: 10.1007/ s10340-019-01089-6
33. Isman, M.B., 2020. Botanical insecticides in the twenty-first century-fulfilling their promise? Annual Review of Entomology, 65: 233-249. doi: 10.1146/annurev-ento-011019-025010

34. Chaudhari, A. K., V.K. Singh, A. Kedia, S. Das and N.K. Dubey, 2021. Essential oils and their bioactive compounds as eco-friendly novel green pesticides for management of storage insect pests: Prospects and Retrospects. Environmental Science and Pollution Research, 28: 18918-18940. doi: 10.1007/S11356-021-12841-W
35. Nenaah, G.E., 2014. Chemical composition, toxicity and growth inhibitory activities of essential oils of three Achillea species and their nano- emulsions against *Tribolium castaneum* (Herbst). Industrial Crops and Products, 53: 252-260. doi: 10.1016/j.indcrop.2013.12.042
36. De Lira, C.S., E.V. Pontual, L.P. de Albuquerque, L.M. Paiva, P.M.G. Paiva and J.V. De Oliveira, 2015. Evaluation of the toxicity of essential oil from Alpinia purpurata Inflorescences to *Sitophilus zeamais* (Maize weevil). Crop Protection, 71: 95-100. doi: 10.1016/j.cropro.2015.02.004
37. Lo´pez, M.D. and M.J. Pascual-Villalobos, 2010. Mode of inhibition of acetylcholinesterase by monoterpenoids and implications for pest control. Industrial Crops and Products, 31: 284-288. doi: 10.1016/j.indcrop.2009.11.005
38. Regnault-Roger, C., C. Vincent and J.T. Arnason, 2012. Essential oils in insect control: low-risk products in a high-stakes world. Annual Review of Entomology, 57: 405-424. doi: 10.1146/annurev-ento-120710-100554
39. De Oliveira, J.L., E.V.R. Campos, M. Bakshi, P.C. Abhilash and L.F. Fraceto, 2014. Application of nanotechnology for the encapsulation of botanical insecticides for sustainable agriculture: prospects and promises. Biotechnology Advances, 32: 1550-1561. doi: 10.1016/j.biotechadv.2014.10.010
40. Garrido-Miranda, K.A., J.D. Giraldo and M. Schoebitz, 2022. Essential Oils and their formulations for the control of curculionidae Pests. Frontiers in Agronomy, 4: 876687.doi:10.3389/fagro.2022.876687
41. Zinhoum, R.A., 2014. Biological study on mediterranean flour moth, *Ephestia Kuehniella* (Zeller) and it's control by environmentally friendly means. Ph. D thesis, Department of Plant Protection, Faculty of Agriculture Moshtohor, Benha University.
42. Pasha, S.S., 2021. Assessment of some essential oils and their mixtures as material repellents against adults of the confused flour beetle, *Tribolium confusum* (Du Val) (Coleoptera: Tenebrionidae), Arab University Journal of Agriculture Science, Ain Shams University., Cairo, Egypt, 29(2): 753-763.
43. Rajashekar, Y., N. Bakthavatsalam and T. Shivanandappa, 2012. Botanicals as grain protectants, Psyche: A Journal of Entomology. Article ID 646740.
44. Singh, P. and A.K. Pandey, 2018. Prospective of essential oils of the genus *Mentha*as bio pesticides: a review. Frontier in Plant Science, 9: 1295. https://doi.org/10.3389/ fpls.2018.01295