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Botanical powders and essential oils alone or carried on Celatom® as rice protectants against the rice weevil *Sitophilus oryzae* (Curculionidae)

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ABSTRACT

This study was conducted to determine the insecticidal activity of four botanical powders and four essential oils (EOs) (alone or carried on a diatomaceous earth compound, Celatom®) against the adults of the rice weevil *S. oryzae*. Different concentrations of each of the tested powders were admixed with two varieties of rice grains (Giza 4000 and Sakha 102) and bioassayed for a week. The results showed that weevil mortality increased with increasing doses of the evaluated plant materials. Pomegranate peel and Lemon grass powders at the lowest tested concentration (0.1g/100g rice grains) caused as high adult mortalities as 76.6 & 65.0 and 73.3 & 60.0% on the two tested rice grains (Giza 4000 and Sakha102, respectively) as compared with the other two tested powders. However, all the tested powders were highly effective as entomocidal materials that elicited complete weevil mortality (100%) at the highest concentration of 2.0g/10g of rice except Marjoram (91.6- 96.6%). Meanwhile, essential oils from four species of plants were obtained by Clevenger-type water distillation and their fumigant toxicities were tested against adults of the rice weevil, *Sitophilus oryzae* (Curculionidae). For assaying the fumigant toxicities of the evaluated EOs, the mortality was determined after 24, 48 and 72 hrs from beginning of exposure and LC50 values of each essential oil were estimated. Fumigation bioassays revealed that essential oils of two plants (Clove and Lemon) had strong insecticidal activity on the experimental insect. Based on LC_{50} values, the order of toxicity of the evaluated EOs to *S. oryzae* from highest to lowest is: Clove (*Syzygium aromaticum*) > Lemon (*Citrus limon*) >Orange (*Citrus aurantium*)>Thyme (*Thymus vulgaris*). Lemon and Orange Eos/Celatom® formulations were the most effective against the rice weevil *S. oryzae*. These two formulations showed the lower LC_{50} after they have been exposed to insects for 7 days. Clove Eos/Celatom® formulation was more toxic than Thyme/Celatom® formulation which has been proved to be the least toxic evaluated formulation.

Keywords: Botanical powders, essential oils, *Sitophilus oryzae*

Introduction

Rice (*Oryza sativa* L.) is one of the world's most important cereal crops providing food for more than one third of the world's population (Younan *et al*., 2011). Stored insect pests are a major problem throughout the world as they significantly reduce the quantity and quality of rice. The rice weevil, *Sitophilus oryzae* (L.) are considered as the most widespread and destructive primary insect pests of stored legumes and cereals (Park *et al.,* 2003). Losses of rice grains ranging from 10 to 20% of overall production due to this insect pest have also been reported (Rajendran and Muralidharan, 2001; Philips and Throne, 2010). Synthetic pesticides have been considered the most effective and accessible means to control insect pests of stored products (Huang and Subramanyam, 2005). These chemicals are associated with undesirable effects on the environment due to their slow biodegradation in the environment and some toxic residues in the products for mammalian health (Benhalima *et al*., 2004; Isman, 2006; Halder *et al*., 2010). The adverse effects of synthetic pesticides have amplified the need for effective and biodegradable pesticides. Natural products are an excellent alternative to synthetic pesticides as a means to reduce negative impacts to human health and the environment.

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Among various kinds of natural substances that have received particular attention as natural agents for insect management are the botanical powders and essential oils. Many plants are rich in secondary compounds with insecticidal activities, especially monoterpenes and their analogues, which are abundant in essential oils of higher plants. These compounds are typically lipophylic, with potential for toxic interference in basic biochemical processes with physiological and behavioral consequences for the insects (Prates and Santos, 2002). Plant materials that are safe for human consumption and have the ability to retain their insecticidal potency overtime should be recommended due to their efficacy against *S.oryzae* (Akinkurolere, 2007; Ileke and Ogungbite, 2014; Dhaniya and Dayanandan, 2016; Vanichpakorn *et al*., 2017; Suleiman *et al*., 2018). Essential oils (EOs) are renewable, non-persistent in the environment and relatively safe to natural enemies, non-target organisms and human beings (Halder *et al*., 2010). Furthermore, the essential oils have been widely used as anti-parasitical, bactericidal, fungicidal, antiviral and insecticidal materials (Sarwar and Salman, 2015). There are numerous investigations on the insecticidal activity of essential oils against the rice weevil *S. oryzae* (Ebadollahi, 2011; Derbalah *et al*., 2012; Brari and Thakur, 2015; Jayakumar *et al*., 2017; Idouaarame *et al*., 2018). The present study is investigating the entomocidal activity of certain botanical powders (admixed with two rice varieties) and essential oils (EOs) (as fumigants) against *S. oryzae* adults. This study will put emphasis on the practice of the tested powders and essential oils (alone or carried on the diatomaceous earth compound Celatom®) as botanical insecticides protectants against the rice weevil, *S. oryzae* to reduce the negative impacts on human health and environment if they have been used as alternative to synthetic pesticides.

Material and Methods

Insect culture

The rice weevil, *Sitophilus oryzae* L. used for this study was obtained from a stock culture in the Laboratory of Plant Protection Department, Faculty of Agric (Saba Basha), Alex. Univ. Clean polished rice was disinfested in the oven at 70^o C for 1 h and thereafter allowed to equilibrate on the laboratory shelf to cool and reabsorb water which had accumulated in the top part of the jar. Two hundred grams of disinfested rice were introduced into 1.5 liters glass jar and the adults resulted from a previous culture were later added and reared on this disinfested rice. The containers were covered with perforated lid and muslin cloth for ventilation and to prevent the escape of insects.

Sample preparation and essential oil extraction

The buds (Clove), leaves (Thyme) or dried peels (Lemon and Orange) (Table 1) were collected or purchased from the local market in Alexandria, Egypt and then they were washed and dried. Firstly, 100g of each sample were air dried at 25°C and submitted for 4 hours to steam-distillation using the Clevenger type-apparatus. The EOs of all dried samples (100g) were isolated by steam-distillation for 3 h, using a Clevenger-type apparatus according to the method recommended by the British Pharmacopoeia (1988). The distillated essential oils (EO) were dried over anhydrous sodium sulphate and then stored in sealed glass vials at 4 - 5°C until use.

Fumigant toxicity by Eos

Vapour toxicity of the tested botanical essential oils against the adult insects of *S. oryzae* was determined via impregnated paper assay following the method of Park *et al.* (2003) with some modifications. Glass jam jars with screw lids were used as exposure chambers. Different doses were applied to a squared circular filter paper (Whatman No. 1) 2cm X 2cm). The treated filter paper was then introduced into the jars to after they are being attached to the inner surface of the screw lid of the jar using a double face adhesive tape. Ten adults of *S. oryzae* (2 to 4 days old) were released into each jar. Insect mortalities were determined and calculated after 24, 48 and 72 hours from exposure. Three replicates were set up for each dose and control.

Efficacy of EOs/Celatom® formulations and botanical powders against *S. oryzae*

1- The tested botanical fine dusts

Certain powder parts of different four medicinal plants (Pomegranate peel, Lemon grass leaves, Marjoram flowers and Chamomile flowers) were used for present investigation (Table 1). The used parts of each medicinal plant were collected, dried in the open air and thereafter, put in an oven at 60° C till complete dryness. Then, these dried parts of each plant were ground by means of electric mill till the attainment of the completely dried fineness. The obtained final powdered parts of each plant species were sieved with 100 mesh sieve to obtain the fine and/or coarse dusts before the application to the grains. Herein, the collected fine tiny particles of sieved ground plant parts, from the pores (100-mesh) of the sieve are considered to be the fine dusts where the particles of the fine powders were about 149 µm (see conversion tables on net: aqua@ecotao.co.za).

Table 1: The evaluated Eos and botanical powders and their used parts of certain plants

Common name of plant	Scientific name	Family	Part used	Major constituents
EOs				
Clove	*Syzygium aromaticum* (Linn.) (Merrill. & Perry.)	Myrtaceae	Buds	Eugenol (71.56 %) and eugenol acetate (8.99 %)[1]
Lemon	*Citrus limon* L.	Rutaceae	Peel	Limonene (93.5%)[2]
Orange	*Citrus aurantium* L.	Rutaceae	Peel	Limonene (89.8% - 94.12)[3&4]
Thyme	*Thymus vulgaris*L.	Lamiaceae	Leaves	1,8 cineole (34.69-40.67) and Linalool (8.99-11.75)[5]
Botanical powders				
Pomegranate	*Punica granatum* L.	Lythraceae	Fruits peels	Flavonoids, ellagitannins and proanthocyanidin compounds [6]
Lemon grass	*Cymbopogon citrates* Stapf	Poaceae	Leaves	75-85% citral [7]
Marjoram (Italian oregano)	*Origanum majorana* L.	Lamiaceae	Leaves	Terpinen-4-ol (26.12%), (Z)-Sabinene hydrate, Linalyl acetate γ-Terpinene (12.96%), α-Terpineol (3.6%) [8&9]
Papong (Chamomile)	*Matricaria chamomilla* L.	Asteraceae	Leaves &Flowers	chamazulene content (1–15%), α-bisabolol and related sesquiterpenes (up to 50% of the oil)[10]

[1]Nassar *et al.,* (2007, [2]Verzera *et al.,* (2004), [3]Camara *et al.,* (2015), [4]Bendaha *et al.,* (2016), [5]Cases *et al.,* (2009), [6]Mirdehghan and Rahemi, (2007), [7]Rauber *et al.,* (2005), [8]Anonymous, (2014), [9]Hosny *et al.,* (2018) and [10]Bruneton, (1995).

2- Preparation of EOs/Celatom® formulations

Each of the evaluated EOs was dissolved in acetone as a suitable organic solvent (1% v/v). One ml of each of these prepared solutions was added slowly to Celtom® (a diatomaceous earth) with constant shake and tilted up and down in a beaker covered with Cling film and left for 24 hrs. Again, the prepared dust was shake and tilted up and down to obtain a homogenous preparation. Finally, the preparation was transferred into stoppered dark glass bottle and shaken to allow random distribution of dust particles.

3- Bioassay of the tested EOs/Celatom® formulations and botanical powders

EOs/Celatom® formulations and fine botanical powders were tested at different weights/100 g rice grains. Three replicates were fixed for each treatment by placing the rice grains (100 g/replicate) in glass jar after sterilizing the rice grains and the glass jars in the oven of at 70 °C for one hour. The grains were then mixed with certain concentrations of the tested powders (fine botanical or EOs/Celatom® formulations) weighed by a sensitive balance. The admixing of rice with the powders was done very well to make sure that the surface of the grain is completely covered. Then, the adult insects (unsexed) were placed at the rate of 50 insects for each replicate. The untreated check (control) was performed without either botanical powders or EOs/Celatom® formulations. The jars were covered by Clingfilm® and the wheat was admixed well with the botanical powders (Singh, 1981; Hose, 1984). The different treatments were left at the lab's conditions for a week. The insects of the experiment were checked after that, and the dead ones were counted and the mortality percentages were recorded for each treatment.

Statistical analysis

All the data concerning mortality were corrected by using Abbott's formula (Abbott 1925). Data of adult mortality were also subjected to probit analysis to determine the lethal concentration required by each EO to kill 50% (LC_{50}) of *S. oryzae* and the other related toxicological parameters (Finney, 1971) after different exposure periods (24, 48 and 72 hrs).

Results and Discussion

The efficacy of powders of Pomegranate peel (*Punica granatum* L.), Lemon grass (*Cymbopogon citrates* Stapf), Marjoram (*Origanum majorana* L.) and Chamomile (*Matricaria chamomilla* L.) as entomocide materials against *Sitophilus oryzae* infesting rice grains was investigated at ambient temperature (25±1ºC) and relative humidity (65±5%). Weevils were exposed to different doses (0.1-2.0g of each plant material /100g of rice grains of two Egyptian rice varieties [Giza 4000 and Sakha102]) and mortality was assessed after 7 days post-treatment. The results (**Table 2**) show that weevil mortality increased with increasing doses of the evaluated plant materials.

Table 2: Mortality percentages of *S. oryzae* adults due to the treatment with different concentrations of certain botanical powders admixed with two different rice varieties for 7 days

Concentration (g/100g Rice grains)	Botanical powders admixed with two rice varieties							
	Pomegranate		Lemon grass		Marjoram		Chamomile	
	Giza 4000	Sakha 102	Giza 4000	Sakha 102	Giza 4000	Sakha 102	Giza 4000	Sakha 102
0.1	76.6 ±0.58	65.0 ±1.00	73.3 ±0.58	60.0 ±1.00	48.3 ±0.60	40.0 ±1.00	45.0 ±1.00	20.0 ±1.00
0.2	81.6 ±0.58	73.3 ±1.00	76.6 ±0.58	83.3 ±3.06	58.3 ±1.53	46.6 ±1.00	68.3 ±0.58	48.3 ±1.53
0.5	90.0 ±1.00	78.3 ±0.58	86.6 ±0.58	85.0 ±1.00	71.6 ±1.16	68.3 ±0.58	83.3 ±1.53	68.3 ±1.53
1.0	96.6 ±1.00	91.6 ±1.53	91.6 ±0.58	95.0 ±1.00	88.3 ±1.53	95.0 ±1.53	100 ±0.00	80.0 ±1.00
2.0	100.0 ±0.00	100.0 ±0.00	95.0 ±1.00	100.0 ±0.00	91.6 ±1.53	96.6 ±1.16	100.0 ±0.00	95.0 ±1.00

Pomegranate peel and Lemon grass powders at the lowest tested concentration (0.1g/100g rice grains) caused as high mortalities as 76.6 & 65.0 and 73.3 & 60.0% on the two tested rice grains (Giza 4000 and Sakha102, respectively) as compared with the other two tested powders. However, all the tested powders (except Marjoram) were highly effective as entomocidal materials that elicited complete weevil mortality (100%) at the highest concentration of 2.0g/10g of rice. Nevertheless, Marjoram caused a highest mortality of 96.6% % when it was admixed with Sakha102 variety. There was no effect of the rice variety on the entomocidal effect of the all tested botanical powders. The presented results show that all the tested powders could serve as alternatives wherewithal to synthetic insecticides for controlling the rice weevil *S. oryzae*. The lemon grass *Cymbopogon citratus* (DC.) Stapf (Poaceae), also known as bee balm, is a species commonly found in many tropical countries, its essential oil is composed mainly of citral, with 70–80%, which has insecticidal activity (Vartak *et al.*, 1994). The study of Uwamose *et al*. (2017) showed that Lemon grass (*C. citrates*) products are promising insecticides and can be used effectively in the management of *Sitophilus oryzae* in storage. KaraKas (2018) found that *C. citratus* extracts was more effective against the granary weevil *S. granarius* adults. Interestingly, the progeny production (F1) was complete by suppressed even in lowest dose. It was concluded that *C. citratus* can be used for the protection of stored wheat from infestations of *S. granarius*. The powder of Lemon grass and its extracts have been reported to have insecticidal potential against the cowpea weevil *Callosobruchus maculatus* (Ojianwuna and Umoru, 2010) and the khapra beetle (*Trogoderma granarium* Everts.) (Coleoptera: Dermestidae) (Asawalam and Igwe, 2012). The results of Al-mousawi *et al*. (2012) revealed that Pomegranate peel powder had repellent and lethal effects against the rice weevil *S. oryzae*. Meanwhile, essential oils (EOs) from four species of plants were obtained by Clevenger-type water distillation and their fumigant toxicities were tested against adults of the rice weevil, *Sitophilus oryzae* (Curculionidae). The mortality determined after different exposure periods of 24, 48 and 72 post rice treatments with different concentrations of the tested EOs. The presented results in Table 3 showed that as the time of exposure increase the weevil mortality increased and the calculated LC_{50} decreased. The results indicated that the obtained EO of Clove (*Syzygium aromaticum*) was the most effective tested EO as compared with the other evaluated EOs. Clove gave low LC_{50} (299.47, 43.65 and 7.453µl/370 ml air) after the different bioassay periods of 24, 48 and 72 post rice treatments, respectively. Clove was followed by Lemon (*Citrus limon*) which gave LC_{50} values of 641.813, 118.612 and 12.268µl/370 ml air. On the other hand, Thyme (*Thymus vulgaris*) was the least effective tested EO as compared with the other tested EOs and it showed its entomocidal activity after 48 and 72 hrs of exposure with high values of LC_{50} (739.558 and 166.733, in respect). Based on LC_{50} values, the order of toxicity of the evaluated EOs to *S. oryzae* from highest to lowest is: Clove (*Syzygium aromaticum*) > Lemon *Citrus limon* > Orange (*Citrus aurantium*) >Thyme (*Thymus vulgaris*). It could be said that both the evaluated botanical powders and EOs of different parts of certain selected plants evoked a high mortality effect on *S. oryzae*. Therefore, this study shows that EOs of *Syzygium aromaticum* and *Citrus limon* could serve also as an alternative to synthetic insecticides in controlling *S. oryzae* infesting rice.

Table 3: Response of the rice weevil *S. oryzae* to different botanical essential oils (LC_{50} values and their corresponding parameters) through fumigant toxicity bioassays (24, 48 and 72 hrs)

Essential oil Biaassay Time (hrs)	**Time**	**LC_{50} (µl/370 ml air)**	**Confidence limits at 95 %**		**Slope ± SE**	**χ^2**
			Lower	**Upper**		
Clove *Syzygium aromaticum*	24	299.47	232.08	570.94	1.114±0.133	19.79
	48	43.65	31.568	59.507	1.289±0.062	70.618
	72	7.453	4.98	10.219	1.475±0.066	78.662
Lemon *Citrus limon*	24	641.813	410.676	1821.04	1.182±0.253	0.905
	48	118.612	89.341	169.852	1.616±0.104	66.474
	72	12.268	8.286	16.892	1.77±0.072	111.223
Orange *Citrus aurantium*	24	374.604	268.268	724.199	0.985±0.183	2.309
	48	76.513	63.551	90.701	2.136±0.127	30.973
	72	25.938	15.77	37.63	2.482±0.104	225.595
Thyme *Thymus vulgaris*	24	--	--	--	--	--
	48	739.558	490.008	1625.148	1.497±0.251	2.82
	72	166.733	144.513	200.436	2.545±0.195	16.727

Plants products are increasingly used in the management of insect pest. They are superior to chemical pesticides because of their bio degradable nature and lesser environmental toxicity. Different types of plants and different plant parts are used in the preparation of plant-based insecticide. Saheb and Mouhouche (2016) found that Clove and Thyme EOs in fumigant test showed the highest efficiency causing 100%mortality of *Sitophilus oryzae* when ther were used at the dose of 240 µl/l. Also, the results of Jairoce *et al.* (2016) showed that the essential oil of Clove caused mortality of 100% 48 h after treatment with the concentrations of 17.9 µl/g. The peel oil has been reported to have toxicity, feeding deterrent and development effects on the rice weevil, *Sitophilus oryzae* (L.) (Tripathi, *et al.,* 2003). Moreover, Orange peel essential oil was also found to have entomocidal effect against *Sitophilus* spp where EO from *C. sinesis* at the highest rate of 750mg/10 ml of acetone applied at 3ml /filter paper gave 100% mortality for *Sitophilus* spp (Kidane, 2005). Fumigant toxicity (expressed in terms of adult mortality) was evaluated by Jayakumar *et al.* (2017) where certain selected plant oils were tested at the concentrations of 10 and 50µl for 24, 48 and 72 hours, and Lemon oil exhibited the highest activity and their respective LD50 values were 58.86, 44.90 and 40.38µl. The evaluated EOs were adsorbed on a commercial diatomaceous earth ((DE) silica-based product (Celatom®). The results of the toxic effect of these essential oils /Celatom® formulations against the rice weevil *S. oryzae* as admixed with two rice varieties (Giza 400 and Sakha102) is shown in Table 4 where different concentrations of the prepared formulations were bioassayed for a period of 7 days. Data for the probit-log dose lines, slope and goodness of fit of each line (measured by χ^2) are also presented. Lemon and Orange Eos/ Celatom® formulations were the most effective against the rice weevil *S. oryzae*. These formulations showed the lower calculated LC_{50} values (mg of the prepared formulation/100g rice grain) of 0.639 &0.798 (Orange) and 0.755 & 0.682 (Lemon) on two rice varieties (Giza 4000 & Sakha 102, respectively).

Table 4: Toxicity of certain essential oils carried on Celatom® against the rice weevil *S. oryzae* as admixed with two rice varieties (Giza 400 and Sakha102)

Essential oils	Rice Variety	LC_{50} (mg/100g rice grain)*	Confidence limits at 95 %		Slope ± SE	χ^2
			Lower	Upper		
Clove *Syzygium aromaticum*	**Giza 4000**	1.44	1.277	1.663	2.486±0.216	6.285
	Sakha 102	2.284	1.836	3.068	1.7±0.164	2.524
Lemon *Citrus limon*	**Giza 4000**	0.755	0.594	0.986	3.135±0.185	31.592
	Sakha 102	0.682	0.527	0.907	3.003±0.177	34.521
Orange *Citrus aurantium*	**Giza 4000**	0.639	0.582	0.704	2.578±0.157	7.717
	Sakha 102	0.798	0.646	1.024	2.094±0.142	14.968
Thyme *Thymus vulgaris*	**Giza 4000**	7.276	4.007	25.601	1.2±0.228	2.146
	Sakha 102	3.553	2.667	5.663	1.959±0.266	2.064

*Mg of the prepared EOs/Celatom® formulations (1 ml of 1% EO acetone solution carried on 100 g Celatom®)

Clove Eos/ Celatom® formulation was more toxic than Thyme / Celatom® formulation which has been proved to be the least toxic evaluated formulation. Celatom® diatomaceous earth (DE) is also known as diatomite or kieselgühr. Celatom® DE has been used against the stored product insect-pests.The entomotoxicity of Celatom® against *T. castaneum* was proved (Al-Besaerawi, 2018). Celatom® is believed to absorb the protective waxy outer layer of insects, leaving them vulnerable to dehydration and death. The possibility of producing Croatian diatomaceous earth (DE) and plant substances (i.e. Thyme and Marjoram) to be used in the near future for controlling *S oryzae*, *R dominica* and *T castaneum* is recently reported by Rozman *et al.* (2018). Therefore, the combination of botanical substances (powders or EOs) and DEs would be effective against sore product insects. It could be concluded that the essential oil technology is a simple applicable and outstanding promising technology and the EOs/Celatom® DE formulations or the combinations of diatoms and plant powders and plant extracts would be effective strategy for controlling the insect-pests of rice grains.

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