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Insecticidal activity of six Apiaceae essential oils against *Spodoptera littoralis* Biosduval (Lepidoptera: Noctuidae)

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Abstract – The African cotton leafworm, *Spodoptera littoralis* Biosduval, is a polyphagous pest, having a wide host range such as many vegetable, fruit and ornamental crops. In this work six Apiaceae essential oils were tested for their insecticidal activity against larval stage of this pest. Essential oils were extracted by hydrodistillation from Tunisian Apiaceae plants: *Carum carvi* L., *Coriandrum sativum L.*, *Cuminum cyminum* L., *Daucus carota* L., *Foeniculu mvulgare* Mill. and *Petroselinum crispum* Mill.. Chemical analysis by GC-MS showed thatThe major compounds were respectively, the carvone (67.6%) and DL-limonene (28.5%), the linalol (77.2%) and the β-myrcene (7.08%), the 2-methyl-3-phenylpropanal (34.2%) and the S-(-)-1-phenyl (23.6%), the β-myrcene (26,9 %) and the elemicin (12,30 %), the trans-anethol (64.1 %) and the L-fenchone (23.3 %), the myristicine (56.1%) and the apiol (16.09 %). Insecticidal bioassay showed that *C. carvi*, *D. carota* and *P. crispum* oils caused mortality higher than 90 % at 200 µl/l air for 24 hours of exposure, however, *C. cyminum* and *F. vulgare* oils had induced 100 % of larval mortality. The determination of the LD_{50} (table 1) showed that *C. carvi* oil seemed to be the most effective oil at 41.45 µl/l air LC_{50}. For *C. sativum*, *C. cyminum*, *D. carota*, *F. vulgare* and *P. crispum*, the LC_{50} was, respectively, 125.87, 64.95, 91.95, 51.22 and 124.31 µl/l air.

Keywords: Caraway, Coriander, Cumin, Carrot, Fennel, Parsley, African cotton leafworm.

1. Introduction

Insect pests present a major constraint in crop production, especially in developing countries (Fan et al. 2011). In order to limit the damages caused by some insects on the hand, and to reduce the overuse of chemical insecticides on the other hand, the insecticidal potential of many plants essential oils were investigated (Elumalai et al. 2010). The Lamiaceae, the Myrtaceae, the Rutaceae and the Apiaceae are some of the most known and used families for their richness of essential oils (Machial 2006). Essential oils extracted from plants can be toxic against insect, especially when they are tested by fumigation (Koulet al. 2008). They also have repellent (Fang et al. 2010), attract (Sharabyet al. 2009) and antifeeding (Pavela et al. 2010) activities against insect pests. These oils can also disturb the insect growth and development and inhibit eggs oviposition and eclosion (Tripathi et al. 2003).

The African cotton leafworm *S. littoralis* causes serious damages on important economic crops such as cotton, tomato and tobacco. The larvae of this pest can feed on 90 economically important plants belonging to 40 families (Azab et al. 2001). Generally, this pest control depends on the use of neurotoxic insecticides, including organophosphates, carbamates and pyrethroids. However, the insect was able to develop resistance toward the majority of these compounds (Abo Elghar et al. 2005). Thus, alternatives for the chemical insecticides based on natural source, such as plants essential oils, are increasingly required.

The present work is aimed to identify the chemical composition of six Apiaceae essential oils and to assess their insecticidal activity against the third instar larvae of *Spodoptera littoralis*, by fumigation tests.

2. Material and Methods

2.1. Plant material

The seeds of *Carum carvi*, *Coriandrum sativum*, *Cuminum cyminum* and *Foeniculum vulgare* were purchased from the same supplier, stating that the origin of these seeds was the Tunisian northwest. For *Petroselinum crispum* seeds, they were supplied by a farmer in the region of Chott Meriem, Sousse, Tunisia. *Docus carota* seeds were collected from the same region during the summer season (2012). All these seeds were kept at room temperature.

2.2. Extraction and analysis of seeds essential oils

The essential oils extraction was performed by hydro-distillation using a Clevenger type apparatus. Distillation lasted between three and four hours, and the oils were kept in a refrigerator at 4°C.

2.3. Chemical analysis of the essential oils

The chemical composition of essential oils was performed by coupling Gas Chromatography with Mass Spectrometry (GC- MS). A gas chromatograph (HP 5890 Series II Plus) was coupled to a mass spectrometer (HP 5972 Series). The sample injection was in splitless mode. The column used was non-polar, type HP- 5MS, its length was 30 m, its internal diameter was 0.25 mm and the film thickness was 0.25 µm. The temperature of the injection was 250°C and the detector temperature was 280°C. The carrier gas was the helium with a flow rate of 1 ml/min and the pressure was 7.7 psi. The column temperature was programmed from 40°C to 250°C at 5°C/min. The injected volume was 1 µl after making a 0.1% dilution for all essential oils except that of *C. carvi* (the dilution was 2%).
The composition was indicated as a relative percentage of total peak area. Spectral analysis of the compounds is carried out by comparison with their counterparts using the spectral library Wiley 7n.l.

2.4. Insect rearing

Spodopetera littoralis was reared under laboratory conditions (25±2°C, 60±10% RH and 16:8 h L:D photoperiod) on an artificial diet for several generations.

2.5. Fumigation test

10 *S. littoralis* third instar larvae (L_3) were placed into a 40 ml volumes cups. A filter paper (Whatman n°1) was fixed on the cups cover after its imbibition with the tested essential oil. For each oil, 4 doses were tested;1, 2, 4 and 8 µl, thus, the tested concentrations corresponded respectively to 25, 50, 100 and 200 µl/l air. Each treatment, in addition to the control test, was repeated 5 times.
The larval mortality was determined after 24 hours of the test completion. A larva was considered dead when it was completely immobile after excitation by a thin needle-nose plier.

2.6. Data analysis

Values were expressed as average of five replicates. The variance analysis was done by one-way ANOVA to $p < 0.05$. Comparisons of averages were performed by Duncan's test using version 18 of the Statistical Package for the Social Sciences program (SPSS).
Lethal Concentrations (LC_{50}) were calculated based on the obtained results of larvicidal effect. LC_{50} values were calculated using probit analysis as described by Finney (1971).

3. Results

3.1. Chemical analysis

The chemical composition of the seeds essential oil was determined by both GC/MS techniques (Table 1). Concerning *C. carvi* oil, the results showed that this oil contained 11compoundspresenting 97.9 % of the total composition. The carvone and d-limonene is the major compounds, with the respective percentages of 67.6% and 28.512%. The other compounds were present at low contents. Among them, the methylbenzoate (0.59%), the anethol (0.41%) and the trans-limonene oxide (0.26%).
For *C. sativum* seeds essential oil, the results showed that it contained 10 compounds representing 97.8 % of the total composition (Table 1). The l-lonalol and the β-myrcene were the major compounds with respective percentages of 77.2% and 7.08%. The other compounds were present at low contents. Among them, the camphor (3.32%), the dl-limonene (3.03%) and the cis-ocimene (2.50%).

Table 1. Mean percentage of major compounds (%) and their retention times (RT) in seeds essential oils

	N°	Compound	RT (min)	% area
C. carvi	**1**	**dl-limonene**	**11.73**	**28.5**
	2	β-ocimene	11.90	0.07
	3	Trans-limonene oxide	14.41	0.26
	4	**Carvone**	**16.34**	**67.6**
	5	Methylbenzoate	19.25	0.59
	6	Anethol	19.35	0.41
	7	2,4,4-trimethyl-4-vinyl-3-cyclopenten-1-one	19.56	0.23
	8	β-elemene	21.75	0.06
	9	Germacrene D	23.87	0.04
	10	Caryophyllene oxide	26.21	0.12
	11	Vulgarol	28.48	0.07
C. sativum	**1**	**β-myrcene**	**10.001**	**7.08**
	2	p-cimene	10.963	1.53
	3	dl-limonene	11.111	3.03
	4	cis-ocimene	11.758	2.5
	5	γ-terpinene	12.036	2.35
	6	**l-linalol**	**13.515**	**77.2**
	7	Camphor	14.588	3.32
	8	Borneol	15.217	0.87
	9	2-methyl-3-phenylpropanal	17.326	tr
	10	carvone	17.437	tr
C. cyminum	1	β-pinene	9.520	8.25
	2	p-cymene	11.000	7.49
	3	γ-terpinene	11.111	10.7
	4	Pulegone	16.031	1.29
	5	**2-methyl-3-phenylpropanal**	**17.529**	**34.2**
	6	2-caren-10-al	18.657	12.26
	7	**S-(-)-1-phenylpropanol**	**18.916**	**23.6**
D. carota	1	α-pinene	8.075	5.24
	2	Sabinene	9.296	3.05
	3	2-β-pinene	9.351	5.63
	4	**β-myrcene**	**9.906**	**26.9**
	5	dl-limonene	11.016	10.4
	6	Cis-ocimene	11.349	4
	7	β-ocimene	11.663	7.34
	8	alloocimene	14.123	2.53
	9	2-methyl-3-phenylpropanal	17.268	2.16
	10	Carvone	17.379	2.94
	11	geranyle acetate	21.189	9.01
	12	**Elemicin**	**25.480**	**12.3**
	13	Italicene	27.552	3.99
	14	Juniper camphor	28.661	2.45
F. vulgare	1	Sabinene	9.335	tr
	2	β-pinene	9.409	tr
	3	β-myrcene	9.927	tr
	4	α-phellandrene	10.278	tr
	5	dl-limonene	11.055	5.225
	6	Cis-ocimene	11.388	tr
	7	**l-fenchone**	**12.868**	**23.3**
	8	Camphor	14.514	tr
	9	Estragol	16.160	3.248
	10	2-methyl-3-phenylpropanal	17.325	tr
	11	**Trans-anethole**	**17.714**	**64.1**
P. cryspum	1	α-pinene	8.206	7.26
	2	β-pinene	9.482	7.65
	3	β-phellandrene	11.092	5.34
	4	α-terpinolene	13.274	tr
	5	(-)myrtenal	16.067	1.99
	6	2-methyl-3-phenylpropanal	17.325	tr
	7	carvone	17.417	tr
	8	**Myristicin**	**24.834**	**56.1**
	9	Elemicin	25.481	1.61
	10	Trans-isomyristicin	26.887	1.84
	11	**Apiol**	**28.422**	**16.09**

For *C. cyminum* seeds oil, the results showed that this oil contained 10 compounds presenting 97.7% of the total composition. The 2-methyl-3-phenylpropanal and the S-(-)-1-phenyl are the major compounds with the percentages of 34.2% and 23.6%, respectively. The other compounds were present at low contents. Among them, the 2-caren-10-al (12.2%), the γ-terpinene (10.7%) and the β-pinene (8.25%). Concerning *D. carota* oil, the chemical analysis results showed that this oil contained 14 compounds presenting 97.9 % of the total content. The major compounds were the β-myrcene and the elemicine with the percentages of 26.9 % and 12.3 %, respectively. Other compounds were also present but at low contents such as the dl-limonene (10.4%), the geranyl acetate (9.01%) and the β-ocimene (7.34%). For *F. vulgare* oil, the results showed that this oil contained 11 compounds presenting 95.8% of the total content. The trans-anethol and the l-fenchone were the major compounds with respectively, 64.1 % and 23.3 %. Other compounds were also present but at low contents such as the dl-limonene (5.22%) and the estragol (3.24%). Concerning *P. cryspum* oil, the chemical analysis showed that this oil contained 11 compounds presenting 97.8% of the total composition. The mysristicin and the apiol were the major compounds with respectively, 56.1% and 16.09 %. Other compounds were also present such as the β-pinene (7.65%), the α-pinene (7.26%), the β-phellandrene (5.34%) and the (-) myrtenal (1.99%).

3.2. Toxicity of the 6 Apiaceae eseential oils against *Spodoptera littoralis* larvae (L_3)

The results of insecticidal bioassay showed that the mortality increased by increasing the doses (Table 2). Mortality rates were low to moderate at 25 µl/l air concentration; they wereranged between 2% and 22%. At 50 µl/l airof *C. cyminum*, *F. vulgare* and *C. carvi* oils, insect mortality was respectively 60 %, 78 % and 94 %. At the highest dose, all oils were able to induce a mortality rate equal or greater than 80%. For *C. cyminum* and *F. vulgare* oils, the mortality reached100 %. The determination of the LC_{50} values showed that the essential oils which were the most toxic against the L_3 of *S. littoralis* were *C. caraway*, with a LC_{50} of 41.451 µl/l air, followed by *F. vulgare* and *C. cyminum* with LC_{50} respectively equal to 51.220 and 64.959 µl/l air. Oils seeds of *D. carota*, *P. crispum* and *C. sativum* caused the less toxic effect against larvae with LC50 equal to 91.950, 124.317 and 125.875 µl/l air, respectively.

Table 2. Mortality pourcentage (%) of *S. littoralis* larvae (L_3) after 24 hours fumigation at different concentrations of essential oils.

Essential oils	Concentrations (µl/l d'air)	Mortality (%)	LC_{50} (µl/l air)
	0	0^a	
C. carvi	25	22^b	41.451
	50	94^c	
	100	96^c	
	200	96^c	
	0	0^a	
C. sativum	25	0^a	125.875
	50	2^a	
	100	62^b	
	200	80^b	
	0	0^a	
C. cyminum	25	4^a	64.959
	50	60^b	
	100	74^b	
	200	100^c	
	0	0^a	
D. carota	25	2^a	91.950
	50	4^a	
	100	72^b	
	200	98^c	
	0	0^a	
F. vulgare	25	12^a	51.220
	50	78^b	
	100	84^b	
	200	100^b	
	0	0^a	
P. crispum	25	2^{ab}	124.317
	50	22^{ab}	
	100	24^b	
	200	92^c	

Alphabetical letters indicates significant difference between concentrations in same insects at P<0.05 (Ducan test). Lethal concentration was calculated with probit analysis method (SPSS).

4. Discussion

Many plant essential oil from Apiaceae Family are well known for their insecticidal activity (Ebadollahi 2013). The essential oil extracted from *C. carvi* seeds at a concentration of 50 µl/l air, was able to ensure a high larval mortality around to 94 %. Thus, this oil seemed to be the most toxic oil against the third instar larvae of *S. littoralis*. In Egypt, the *Coriandrum Sativum* seeds essential oils have important ovicidal activity against *S.littoralis* (khedr and kawas 2013). Otherwise, many studies had been conducted showing the sensitivity of this pest with various plants essential oils. Thus, the essential oil of *Citrus aurantium* was toxic, by fumigation, against the larvae (L_3) of *S. littoralis* at 200 µl/l air after 24 hours of exposure, mortality was total and the LC_{50} was 79.95 µl/l air (Laarif et al. 2013).

Moreover, essential oils from *Salvia officinalis* leaves, *C. sativum* seeds, *F. vulagre* seeds, *D. carota* flowers and *Origanum majorana* leaves had caused high mortality against *S. littoralis* larvae (L_3) by fumigation. LC_{50} were 23.050 µl/l air; 68.925 µl/l air; 95.075 µl/l air; 99.300 µl/l air and 100.925 µl/l air, respectively (Souguir et al.2013). The essential oils of *Thuja occidentalis*, *Tanacetum parthenium* and 8 Lamiaceae, namely, *Origanum vulgare*, *Mentha citrata*, *Nepeta cataria*, *Salvia sclarea*, *Origanum compactum*, *Melissa officinalis*, *Thymus mastichina* and *Lavandula angustifolia* are also toxic by topical application, the LD_{50} were less than or equal to 0.05 µl/larva (Pavela 2005). It is reported that this toxicity was due to the presence of two terpenic substances, namely, camphor and trans-acetate chrysanthenyl, knowing that camphor is the main compound found in some essential oils extracted from aromatic plants like *Eucalyptus* sp. *Cinnamomum camphora*, *Rosmarinus officinalis*, *Arthemisia* sp and *C. caraway*, which may cause mortality of *S. littoralis* larvae (L_3) (Pavela et al. 2010) . The Fresh and dry aerial parts of *Foeniculum vulgare* showed an important insecticidal potential on *Spodoptera littorali*s larvae (Pavela et al 2016). Otherwise, the essential oil extracted from *Tanacetum parthenium* had antifeeding effect on fourth instar larvae of *S. littoralis*. The dose caused 50 % of anti-feedancy (DD_{50}) was 0.25 µl/cm^2. This oil was also able to stop the growth of the *S. littoralis*L_5; and the dose caused 50 % of this effect was 0.53 µl/g (Pavela et al. 2010). Many other essential oils was shown to be active on *S.littorlis* larvae as *Slavia officinalis* (Ben Khedheret al.2017; Reguez et al. 2013, 2018), *Thymus algeriensis* (Belhaj-Ali et al. 2015), *Artemisia absintium* (Dhen et al. 2014, Chaieb et al. 2018), *Eugenia caryophyllata* (Dhen et al. 2013) and Citrus species (Zarrad et al. 2013, Chaieb et al. 2017). In addition, Jacobson (1990) studies showed that the carvone, one of the major compounds of *C. carvi* oil, incorporated at a percentage of 1 % into the diet of *S. littoralis* larvae, caused the decrease of the larvae average weight and blocking adults emergence. Only 2.5 % of treated larvae are transformed into chrysalis. Besides, other studies showed that the seeds essential oil of *C. carvi* was toxic by fumigation against the adults of *Sitophilus zaemais* and those of *Tribolium castaneum*. The CL_{50} were respectively 3.37 and 2.53 mg/l. This oil had also a repellent activity for *Sitophilus oryzae* adults (Fang et al. 2010). Moreover, *C. cyminum* seeds essential oil was toxic against *Sitophilus oryzae* adults when it was tested by 24 hours fumigation. The LC_{50} was 0.67 µl/insect. Essential oil caused the insect death by the inhibition of the AChE activity (Zarrad et al. 2015, 2017a). It was also significantly repellent for these adults (Chaubey, 2011). Further, the topical application of the *F. vulgare* oil was toxic against *S. zaemais*. At 0.75 µl/insect, the mortality was 77 and 98 % after respectively, 24 and 96 hours of exposure (Rossi et al. 2012). In addition, Elumalai et al. (2010) studies showed that *C. sativum* and *C. cyminum* essential oils caused 100 % of antifeeding on *S. litura* $L_4$when they were tested at 6 mg/cm^2, during 24 hours. The active compounds present in the oils have a strong antifeeding activity on larvae.

Finally the essential oils of Apiaceae family offers a source of natural insecticidal substances against *Spodoptera littoralis*, the application of these essential oils can be done in closed area as greenhouses. These essential oil can be also formulated to limit their volatility by fixing these substances on inert powder as clays (Khaled et al. 2017, Zarrad et al. 2017b)

5. Conclusion

Based on all these results, it appears that the use of Apiaceae essential oils in crops protection is a promoted alternative to chemical insecticides overuse and their drawbacks on environment and human health. Thus, the essential oils of six Apiaceae species used in this work could be a potential source of bioinsecticides. *C.carvi* shoes hight toxicity to *Spodoptera littoralis* larva and seems to be an alternative solution for noctuid management in greenhouses. Nevertheless, further researches have to be done, to formulate adequate preparations for insect management and the control of suitable application conditions of theses formulations.

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