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Chemical Composition and Insecticidal Activity of *Mentha rotundifolia* L. Essential Oil against *Sitophilus granarius*

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Original Research Article

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Abstract: In order to contribute to the development of an integrated pest management strategy for stored grains, the insecticidal activity of *Mentha rotundifolia* L. essential oil was evaluated against *Sitophilus granarius*, wheat weevil. Insects and toxicity tests were carried out under laboratory conditions at a temperature of 27 ± 2 ° C and a relative humidity of 60 ± 5%, at Çankırı Karatekin University, Yapraklı Vocational School, Department of Animal and Plant Production. The essential oil was analyzed by gas chromatography (GC) and gas chromatography/mass spectrometry (GC/MS) to give 14 compounds in the essential oil of *M. rotundifolia*. The major component of the oil was identified as 3-Cyclopenten-1-one, 2-hydroxy-3-(3-methyl-2-butenyl)- (89,09%). *M. rotundifolia* essential oil has remarkable insecticidal properties. Highest contact toxicity (100%) of the oil was observed against *S. granarius* (1µL/insect application of 4% (v/v) oil solution in acetone, after 24 h). Highest fumigant toxicity (100%) was observed for *S. granarius* (10µL/10mL container application of 1% (v/v) oil solution in acetone, after 24 h). The set of knowledge elements obtained should help guide strategies for the integrated protection of the grain quality during the storage of local cereals production.

Keywords: Essential oil, *Mentha rotundifolia*, *Sitophilus granarius*, contact toxicity, fumigant toxicity, stored products.

INTRODUCTION

In Algeria, cereals have strategic importance because it is at the base of the country's food security [1]. Cereal grains have always been the main food source for humans and domestic animals.

The knowledge of the phenomena governing their conservation and the control of their storage techniques are decisive for the survival of the world's population which records growth rates barely conceivable, moving humanity from 1.5 billion people around 1850 to more than 7 billion today [2]. The storage conditions, the physiological characteristics of the grain, type of cereal and grain cereals varieties and their origins, contributed significantly in the degree of infestation, species richness and the level of presence of insects [3]. In the case of wheat, these insects can cause considerable losses, up to 50% [4, 5].

The wheat granary weevil *Sitophilus granarius* L. is one of the most widespread and destructive primary insect pests of stored cereals such as wheat, maize, rice, oat, sorghum and barley [6]. In addition to the physical damage that it causes to grain, this insect also allows the entry of pathogenic organisms, such as fungi or bacteria [7]. *S. granarius* adults can survive unfavorable conditions in the structure of storage facilities for some time [8].

Nowadays, insect control strategy in stored-food products relies heavily upon the use of gaseous fumigants and residual insecticides, both of which pose serious hazards to warm-blooded animals and the environment [9], also generated the problem of resistance to phosphine, malathion, pyrethroids, methoprene, hydroprene [10-13].

In recent years, there is an increasing attention on the control of storage pests by natural products, including plant extracts, essential oils and their pure components [14]. Their use has been the subject of several research projects in the last decade and aroused a strong scientific interest reflected in the number of works dealing with the effectiveness of essential oils in the protection of grains and stored products [15-22].

The toxicity of a large number of plant extracts, essential oils and their constituents has been assessed against *S. granarius* [23-30, 9]. Large number of studies involving insecticidal activity of *Mentha* species (Repellent properties, Fumigation activity, antifeedant activity,….) against adult of storage pests were reported [31].

Since it was not done before, the aim of this work is to evaluate the insecticidal activity of *Mentha rotundifolia* essential oil against *Sitophilus granarius*, a well-known pest that causes an economically important yield loss in storage silos.

MATERIALS AND METHODS

Plant material and essential oil extraction

The samples of *M. rotundifolia* used in our study were collected on June 2014 from Chouf Lakdad region (Setif, Algeria). A sample of 100 g of the air dried plant was subjected to hydrodistillation for 3h, using a Clevenger type apparatus [32] to produce essential oil. *M. rotundifolia* essential oil was preserved in an amber flask at low temperature (4°C) until further analysis.

Gas chromatography–mass spectrometry analysis (GC/MS)

The essential oil performed on Agilent Technologies GC 7890A with a built-in 5975 Triple Axis Detector MS system, equipped ionization system, HP5-ms (30 m x 250 mm x 0.25 mm) column and ionization energy (70 eV) for GC-MS detection. The helium was used a carrier gas at a flow rate (1 $ml.min^{-1}$). The same column temperate was achieved with GC analysis given above in our laboratory, by comparison of their mass spectral fragmentation patterns (WILLEY and NIST database/Chem Station data system) and their retention indices (determined with reference to homologue series of normal alkanes).

Insect culture

S. granarius cultures were reared in laboratory at 27±2 ◦C and 60±5 r.h. in a dark climate chamber in the Yapraklı Vocational School, Department of Animal and Plant Production, at Çankırı Karatekin University. The food medium used was wheat for *S. granaries* and 1-2 month-old adults were used in trials. Tests are also carried out in under the same condition and the same laboratory.

Insecticidal activity

Insecticidal contact toxicity assay of essential oil on *S. granarius*

The *M. rotundifolia* essential oil was diluted with acetone to obtain 4%, 2%, 1%, 0,75% and 0,50 (v/v) doses. The essential oil was applied at the rate of 1 µl/insect with a 50 µl Hamilton syringe [33]. Controls were determined using acetone. The essential oil was applied topically to the dorsal surface of thorax of the insects [34]. The treated insects were transferred to the 60 mm diameter Petri dishes. The Petri dishes were incubated at 25±2 ºC and the number of dead insects was recorded after 24 h. For each replication, 10 insect were used and each experiment was repeated five times.

Fumigant insecticidal toxicity assay of essential oil on *S. granaries*

The *M. rotundifolia* essential oil was diluted with acetone to obtain 1%, 0,50% and 0,25 (v/v) doses. In the experiments were used glass tubes (10ml). Adults of *S. granarius* were transferred to glass tubes. The discs were cut 1 cm diameter from filter paper. The stock solutions of oils (10µl) were impregnated to these discs with micropipette. Controls were determined using acetone. The discs were left 5 min. open to evaporate the acetone. The discs were attached to the rubber caps of the test tubes with a needle and caps were closed. The glass tubes were incubated at 25±2 ºC and the number of dead insects was recorded after 24 h. For each replication, 10 insect were used and each experiment was repeated three times.

Statistical analysis

The activity results obtained in the insecticidal activity assays were transformed into percent death values; which were used to obtain the arcsin values [35]. The arcsin values were subjected to variance analysis (ANOVA) followed by Tukey's multiple comparison test with $P < 0.05$ significance level. All of the statistical analysis were performed with Minitab Release 14 program [36].

RESULTS

The essential oil composition of *M. rotundifolia* was given in Table 1. Fourteen compounds were identified. The 3-Cyclopenten-1-one, 2-hydroxy-3-(3-methyl-2-butenyl)- is the major constituent of this oil with a rate of about 89.09%. The remainder consists of monoterpenes and sesquiterpenes.

The examination of Table 1 revealed the presence in *M. rotundifolia* essential oil of certain identical compounds (β-Pinene). Two minor compounds with levels not exceeding 0.5% of the total essential oil were identified: 1-Octen-3-ol, acetate (0.29%) and Borneol (0.38%).

Table-1: Chemical composition of *M. rotundifolia* essential oil

Compound number	RT (mn)	RI	%	Name
1	11.751	907	1.18	α-Pinene
2	12.951	947	0.54	β-Pinene
3	13.135	953	1.17	β-Pinene
4	13.337	959	0.68	β-Pinene
5	14.749	1001	1.74	β-Terpinyl acetate
6	17.304	1077	0.29	1-Octen-3-ol,acetate
7	19.619	1144	0.38	Borneol
8	22.873	1237	0.52	Carvone oxide
9	**26.509**	**1346**	**89.09**	**3-Cyclopenten-1-one, 2-hydroxy-3-(3-methyl-2-butenyl)-**
10	27.352	1372	0.77	3-Methyl-2-pent-2-enyl-cyclopent-2-enone
11	27.548	1378	1.25	Cinerolone
12	28.212	1397	0.91	Caryophyllene
13	28.867	1419	0.70	β-Farnesene
14	30.078	1458	079	β-Cubebene

The results in Table-2 show that the contact insecticidal activity of *M. rotundifolia* essential oil against *S. granarius* revealed a very interesting biological activity. At 4% concentration of this oil, insect mortality is marked by 100% after 24 hours of incubation.

Table-2: Contact activity of *M. rotundifolia* essential oil against *Sitophilus granarius* after 24h of incubation

Doses %	Mortality±StDEV %
Control	0.00±0.00
4	100.00±0.00
2	68.46±1.46
1	62.05±0.22
0.75	29.52±1.72
0.50	0.00±0.00

(F=328,11; d.f.=5,24; P=0.000)

The analysis of Table 3 shows a very interesting fumigant insecticidal activity of *M. rotundifolia* essential oil with a rate that exceeds 80% of mortality at 0.5% concentration and 100% of mortality at 1% concentration, after 24 hours of incubation.

Table-3: Fumigant activity of *M. rotundifolia* essential oil against *Sitophilus granarius* after 24h of incubation

Doses %	Mortality±StDEV %
Control	0.00±0.00
1	100.00±0.00
0,5	83.64±0.67
0,25	0.00±0.00

(F=1158,24; d.f.=3,8; P=0.000)

DISCUSSION

With the 3-Cyclopenten-1-one, 2-hydroxy-3-(3-methyl-2-butenyl)- as major component, the essential oil of our study does not exhibit the same chemical characteristics of the essential oils of *M. rotundifolia* of Algerian origin: as the major compound, piper tone oxide (72.87%) [37], pulegone (70.4%) [38], piperitoneone (33.06%) [39], piper tone oxide (Miliana: 31.4%, Rouina: 19.7%) and piperitenone oxide (Miliana: 27.8%; Rouina: 29.4%) representing chemo type 1[40]. For the sample from Chlef, the major components are piperitenone (54.9%) and piperitenone oxide (17.6%) representing chemo type 2. While the piperitenone oxide was found with 38.6% in Rouina and 23.5% in Miliana [41].

Previous studies in other countries on the chemical composition of the essential oil of *M. rotundifolia* have revealed the existence of chemo types with different major components, such as β-Caryophyllene (26.67%) in Beja and pulegone (32.09%) in Bizerte [42], 2.4 (8), 6-p-menthatrien-2.3-diol in Cuba [43], linolool (35.32%) in Egypt [44], piperitol (57.6%) in Spain [45], Pulgone (85%) [46], Menthol (40.50%) [47] in Morocco. Piperitenone oxide has been reported as a characteristic constituent of volatile oils of *M.*

rotundifolia chemo types with 80.8% in Uruguay [48] and 81.5% in Germany [49].

Despite the large number of chemical types listed in the literature for essential oils of *M. rotundifolia* L., the predominant presence of 3-Cyclopenten-1-one, 2-hydroxy-3- (3-methyl-2-butenyl) - of our oil has never been described, which gives a certain originality to this sample.

Differences in chemical composition observed for essential oils is likely related to abiotic factors such as climate specific regions of provenance samples, geographical factors such as altitude and soil type [40].

According to [42], genetic factors should not be excluded in explaining the chemo-variation of essential oils. Moreover, chemical differences in the oil composition of plant species in relationships with harvesting season were reported [50].

For insecticidal contact activity and after application of the essential oil, we note very high mortality rates that can vary from 60 to 100% depending on the concentration (between 1 and 4%), which is probably due to the synergistic effect between the majority compound and /or the one of the elements present in the composition of this oil.

In general, the toxicity of essential oils isolated from plant samples against stored-product pests is related to their major components [26]. The insecticidal activity against *S. granarius* of the essential oil of several plants was evaluated [51-53, 25-27, 9, 29]. Indeed, the essential oil of *M. rotundifolia* contains mainly hydrocarbon monoterpenes such as α-pinene whose insecticidal properties have already been demonstrated and oxygenated monoterpenes such as borneol, cinerolone whose insecticidal properties have also already been demonstrated against several insects.

However, it would be difficult to think that the insecticidal activity of the oil is limited only to some of its major constituents; it could also be due to certain minority constituents or to a synergistic effect of several constituents [54]. The toxic effects of essential oils depend on the insects species, the time of exposure and the plant [55]. For this last point, the essential oil of *Crithmum maritimum* gave low contact activity against *S. granaries* [30].

Fumigant insecticidal activity, shows that essential oils are sources of biologically active vapors that are potentially effective insecticides [26]. Therefore, the possibility of using these natural fumigants to control insects in stored products may be worthy of further investigation [56].

Among the components of the essential oil, monoterpenoids have attracted the greatest attention for their fumigant activity against insects of stored products [57]. Several reports indicate that monoterpenoids cause insect mortality by inhibiting the activity of acetylcholinesterase enzyme (AChE) [58]. Therefore, it is suspected that, in addition to inhibition of AChE, monoterpenes may also act on other vulnerable sites (for example monooxygenases dependent on cytochrome P450) [59].

The toxicity of the essential oils against the insects of the stored products is influenced by the chemical composition of the oil, depending on source, season and ecological conditions, extraction method, extraction time and plant part used [60].

It is well known that for fumigants, the active stages (adults and non-diapausing larvae) of insects are more susceptible than the sedentary stages (eggs and pupae) due to differences in their respiratory rate [57].

Laboratory tests with plant products in the presence of a stored product such as wheat proved less effective than in the absence of the stored product, mainly due to high sorption of the oil/components by the commodity [61].

In conclusion the essential oil obtained from *M. rotundifolia* afforded high contact and fumigant insecticidal activity against to the major stored product insects namely *S. granarius.* The rapid action of the oil in inducing as much as 100 % mortality within a 24 hour exposure period indicated that it could be a promising fumigant agent against the indicated stored product pest. Considering the results of the contact and fumigant insecticidal activity in this study, the essential oil of *M. rotundifolia* has the potential to be employed in the control of *S. granarius*.

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