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Chemical composition and insecticidal properties of *Lantana camara* L. leaf essential oils from Algeria

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Essential oils extracted from *Lantana camara* were tested for fumigant activity against *Sitophilus granarius* adults. Composition of *L. camara* essential oil included large amounts of sesquiterpene hydrocarbons, mainly β -caryophyllene. The bioactivity of the essential oil extracted by hydrodistillation from leaves of *L. camara* was assessed under laboratory conditions. With fumigation bioassays, essential oils showed different activities on *S. granarius*. April essential oil, after 24 hours of exposure, exerted the highest activity. Similar results were obtained for February and June essential oils after 48 hours of exposure, although December essential oil showed good fumigant activity after 96 hours of exposure. The persistence of the insecticidal efficiency of the essential oil (remanence tests) against *S. granarius* adults confirmed that June essential oil was efficient for 2 weeks. Even if laboratory bioassays are only the first step towards the use of essential oils in practical applications, these substances represent a possible alternative to chemical insecticides in some market places.

Keywords: *Lantana camara* L; essential oil; *Sitophilus granarius*; fumigant activity.

Introduction

Lantana camara L., belonging to the *Verbenaceae* family, is planted as an ornamental (1–3), but it is a highly invasive weed in many parts of the world (4–9). *Lantana camara* is listed as one of the important medicinal plants of the world (10) and is considered one of the most noxious weeds (11, 12). The essential oil showed a wide spectrum of antibacterial, antimicrobial and antifungal activities (13–15). Moreover, the plant has been shown to have toxic and repellent effects against certain insects (16–18).

Studies on plant essential oils and their constituents as fumigants against stored product insects have been reviewed (19). *Sitophilus* species are major pests of stored grains and grain products throughout the world (20). The control of these insects relies heavily on the use of synthetic insecticides and fumigants, which has resulted in several problems, including environmental disturbances, increasing costs of application, pest resistance to pesticides and lethal effects on non-target organisms, in addition to direct toxicity to users (21).

Lantana camara essential oils from different origins have been previously investigated (22–24), showing a great variation in chemical composition. The majority of published works indicate a high content of sesquiterpene hydrocarbon compounds (25). Samples from

Madagascar show that sabinene is the major monoterpene. Among the oxygenated components, davanone is the main one (23).

In the present work, the composition variation of essential oils obtained from *L. camara* were determined. We have undertaken an investigation with fifty-one samples collected every week for 3 years to follow the chemical composition changes during seasons. Essential oils were obtained, taking into account the yellow–orange color of the flower. Hence, the objective of the current study was to determine the chemical constituents and fumigant toxicity of the leaf essential oil of *L. camara* against adults of *Sitophilus granarius*, an important stored-product beetle observed in grain storage facilities in different countries.

Experimental

Plant material

The aerial part of *L. camara* was collected every week during 2007. Six groups were considered: February, April, June, August, October and December. Three other groups are considered at March (2007, 2008 and 2009). The plants were collected from the production area of the botanical garden of the University of Sciences and Technology Houari Boumediene (Bab Ezzouar, a suburb of

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the city of Algiers, coordinates: 36°43'N 3°11'E). Air-dried material (3 kg per group) was hydrodistilled for 3 hours using a Clevenger-type apparatus. Oils were dried over anhydrous Na₂SO₄ and then were kept in sealed vials at 6°C until analysis.

Gas chromatography

The analyses of the volatile compounds were carried out on a Hewlett Packard gas chromatograph–mass

spectrometer (GC–MS) system (GC 6890; MSD 5973). An HP-5 MS capillary column (30 m, 0.25 mm, 0.25 µm film thickness) was directly coupled to the mass spectrometer. The carrier gas was helium (1 mL/minute). The program used was 8 minutes isothermally at 40°C, then 40° to 250°C at a rate of 2° C/minute then held isothermally for 5 minutes. The injection port temperature was 250°C. The ionization of the sample components was performed in the elec-

Table 1. Percentage composition of yellow-orange Algerian *Lantana camara* leaf essential oils during 2007.

No.	Component ^a	RI ^b	RI ^c	Feb.	Apr.	June	Aug.	Oct.	Dec.	Mean	SD
1	2-Hexanal	857	846	tr	tr	tr	tr	tr	tr	–	–
2	α-Thujene	921	924	tr	tr	tr	tr	tr	tr	–	–
3	α-Pinene	926	932	0.8	0.3	0.8	0.2	0.6	0.1	0.5	0.3
4	Camphene	932	946	tr	tr	tr	tr	tr	tr	–	–
5	Sabinene	967	969	1.3	2.4	1.4	2.7	2.1	1.4	1.9	0.6
6	β-Pinene	973	974	0.7	1.3	0.2	1.4	0.8	0.9	0.9	0.4
7	1-Octen-3-ol	978	974	tr	0.8	0.2	0.8	tr	tr	0.3	0.3
8	Limonene	1021	1024	0.2	0.3	0.1	0.3	0.3	0.3	0.2	0.0
9	γ-Terpinene	1061	1054	tr	tr	tr	tr	0.1	0.1	–	–
10	<i>trans</i> -Sabinenehydrate	1092	1098	tr	tr	tr	tr	tr	0.1	–	–
11	Linalool	1098	1098	0.2	0.7	0.5	0.6	0.5	0.4	0.5	0.1
12	Borneol	1157	1165	tr	tr	tr	tr	tr	tr	–	–
13	4-Terpineol	1170	1174	tr	tr	0.1	0.3	0.6	0.6	0.3	0.2
14	α-Terpineol	1184	1186	tr	tr	tr	tr	tr	tr	–	–
15	Persilphiperfol-7-ene* ¹	1336	1336	0.1	0.2	0.1	0.2	0.1	0.3	0.1	0.0
16	Phenol-2-methoxy-3-(2-propenyl)	1345	–	tr	0.1	tr	0.1	0.1	0.1	–	–
17	Caryophylline-(I3)	1368	–	0.4	tr	0.2	0.2	0.2	0.2	0.2	0.1
18	β-Cubebene	1373	1387	0.8	0.2	0.6	0.7	0.8	0.7	0.6	0.2
19	β-Elementene	1376	1389	6.3	6.1	7.2	6.5	3.3	5.3	5.8	1.3
20	Di- <i>epi</i> -α-Cedrene	1389	–	0.1	0.1	0.3	0.2	0.2	0.4	0.2	0.1
21	β-Caryophyllene	1413	1408	26.3	46.7	34.4	40.7	27.0	37.0	35.4	7.9
22	α-Humulene	1439	1452	4.3	3.5	3.7	3.9	4.7	4.3	4.1	0.4
23	Aromadendrene	1443	1439	0.7	0.4	0.2	0.3	0.3	0.3	0.4	0.2
24	β-Farnesene	1446	1454	1.1	0.8	0.4	1.1	0.9	1.0	0.9	0.2
25	<i>trans</i> -Muurolo-3,5-diene	1453	1453	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.0
26	α-Acoradiene* ²	1468	1466	15.3	10.2	4.4	5.2	6.1	3.7	7.5	4.4
27	α-Curcumene	1475	1479	1.6	4.1	4.2	3.3	3.2	4.7	3.5	1.0
28	Germacrene D* ³	1484	1484	4.3	2.5	3.3	4.3	2.5	2.6	3.2	0.8
29	α-Zingiberene	1487	1493	1.9	2.1	0.2	2.9	2.6	3.5	2.2	1.1
30	β-Guainene* ⁴	1491	1492	0.5	2.3	1.1	0.8	0.6	0.7	1.0	0.6
31	γ-Cadinene	1514	1513	2.6	1.3	4.1	4.4	3.6	4.9	3.5	1.3
32	α-Cadinene	1520	1537	1.1	0.9	0.1	0.1	0.3	0.1	0.4	0.4
33	Caryophyllene oxide	1573	1582	12.4	2.2	10.9	5.0	18.8	6.8	9.4	5.9
34	β-Himachalene oxide* ⁵	1617	1616	0.2	0.1	0.1	tr	0.4	0.3	0.2	0.1
35	<i>epi</i> -bicyclosquiphellandrene* ⁶	1623	–	0.1	tr	0.5	0.9	0.9	0.9	0.6	0.4
36	1- <i>epi</i> -Cubanol* ⁷	1630	1628	1.6	1.2	3.3	2.4	3.6	4.3	2.7	1.2
37	γ-Eudesmol	1634	1632	0.4	0.2	0.7	0.5	0.6	0.8	0.5	0.2
38	<i>epi</i> -α-Cadinol	1643	1638	1.2	0.6	1.9	1.2	1.6	2.1	1.4	0.5
39	Isoaromadendrene epoxide	1656	–	tr	tr	0.1	0.1	0.5	tr	0.1	0.2
	Yields (g/kg)			1.4	1.5	1.1	1.0	1.1	1.2	1.2	0.2
	Total listed (%)			88.6	93.3	87.3	93.3	89.6	90.9	90.5	2.5

Notes: ^aCompounds listed in order of elution from an HP-5 MS capillary column. RI^b, retention indices relative to *n*-alkanes C₇–C₂₇ on non-polar column HP-5 MS; RI^c, retention index on DB-5 (30), tr: trace (<0.1%). *Identification tentative. ¹*m/z* (% rel. int.): 100 (119), 96 (105), 95 (161), 47 (91), 29 (81), 25 (93), 23 (41), 22 (204), 20 (120), 20 (92), (79 *m/z*). ²*m/z* (% rel. int.): 100 (119), 76 (121), 58 (93), 45 (204), 44 (105), 38 (91), 27 (41), 22 (79), 21 (77), 20 (107), (119 *m/z*). ³*m/z* (% rel. int.): 100 (91), 98 (69), 92 (77), 81 (105), 43 (94), 38 (120), 36 (56), 33 (204), 30 (121), 25 (161), (99 *m/z*). ⁴*m/z* (% rel. int.): 100 (159), 93 (105), 85 (131), 75 (220), 69 (177), 69 (91), 68 (202), 52 (106), 47 (144), 40 (96), (27 *m/z*). ⁵*m/z* (% rel. int.): 100 (136), 74 (79), 73 (91), 54 (95), 46 (107), 40 (161), 39 (135), 37 (92), 33 (43), 31 (105), (86 *m/z*). ⁶*m/z* (% rel. int.): 100 (119), 46 (204), 30 (162), 27 (105), 26 (121), 19 (189), 15 (134), 12 (95), 12 (81), 11 (133), (91 *m/z*). ⁷*m/z* (% rel. int.): 100 (95), 81 (121), 40 (43), 38 (204), 38 (164), 35 (109), 28 (94), 26 (71), 26 (79), 17 (69), (131 *m/z*).

tron ionization (EI) mode (70 eV). The linear retention indices for all the compounds were determined by injection of the sample and a solution containing the homologous series of C₇–C₂₇ *n*-alkanes into the GC–flame ionization detector (GC–FID) under the same conditions, identical to GC–MS conditions. The individual constituents were identified by their identical retention indices, referring to those obtained from literature (22, 25–28), and by comparing their mass

spectra with either the known compounds or with the MS data bank.

Insect cultures

Sitophilus granarius was reared in 1-L glass jars containing chickpea grains, which were covered by fine mesh cloth for ventilation. Adult insects, 1–7 days old, were used for the fumigation toxicity test. All experimental procedures were conducted under

Table 2. Percentage composition of yellow-orange Algerian *Lantana camara* leaf essential oils at March over 3 years.

No.	Component ^b	RI ^a	RI ^c	2007	2008	2009	Mean	SD
1	2-Hexanal	857	846	tr	tr	tr	–	–
2	α -Thujene	921	924	tr	tr	tr	–	–
3	α -Pinene	926	932	0.2	0.1	0.2	0.2	0.0
4	Camphene	932	946	tr	tr	tr	–	–
5	Sabinene	967	969	4.0	2.4	1.5	2.6	1.2
6	β -Pinene	973	974	2.1	1.1	0.6	1.3	0.7
7	1-Octen-3-ol	978	974	tr	0.4	0.6	0.3	0.3
8	Limonene	1021	1024	0.4	0.2	0.1	0.3	0.1
9	γ -Terpinene	1061	1054	tr	tr	tr	–	–
10	<i>trans</i> -Sabinenehydrate	1092	1098	tr	tr	tr	–	–
11	Linalool	1098	1098	0.7	0.7	0.6	0.7	0.0
12	Borneol	1157	1165	tr	tr	tr	–	–
13	4-Terpineol	1170	1174	0.5	0.4	0.2	0.4	0.1
14	α -Terpineol	1184	1186	0.2	0.2	tr	0.1	0.1
15	Persilphiperfol-7-ene* ¹	1336	1336	0.2	0.1	0.1	0.1	0.0
16	Phenol-2-methoxy-3-(2-propenyl)	1345	–	tr	0.1	tr	–	–
17	Caryophylline-(13)	1368	–	0.1	0.1	0.2	0.1	0.0
18	β -Cubebene	1373	1387	0.4	0.4	0.4	0.4	0.0
19	β -Elemene	1376	1389	6.2	6.3	6.4	6.3	0.0
20	Di- <i>epi</i> -Cedrene	1389	–	tr	tr	tr	–	–
21	β -Caryophyllene	1413	1408	47.1	46.3	36.7	43.4	5.8
22	α -Humulene	1439	1452	3.2	3.6	3.7	3.5	0.2
23	Aromadendrene	1443	1439	0.8	0.3	0.3	0.4	0.2
24	β -Farnesene	1446	1454	0.8	0.8	0.8	0.8	0.0
25	<i>trans</i> -Muurolo-3,5-diene	1453	1453	0.1	0.1	0.1	0.1	0.0
26	α -Acoradiene* ²	1468	1466	3.2	9.3	9.7	7.4	3.6
27	α -Curcumene	1475	1479	3.2	1.1	1.3	1.8	1.1
28	Germacrene D* ³	1484	1484	2.4	3.8	2.8	3.0	0.6
29	α -Zingiberene	1487	1493	3.1	3.4	3.8	3.4	0.3
30	β -Guainene* ⁴	1491	1492	1.2	3.1	2.7	2.3	0.9
31	γ -Cadinene	1514	1513	1.3	4.1	4.2	3.2	1.6
32	α -Cadinene	1520	1537	0.1	0.1	0.1	0.1	0.0
33	Caryophyllene oxide	1573	1582	3.6	3.4	9.8	5.6	3.6
34	β -Himachalene oxide* ⁵	1617	1616	tr	tr	0.1	–	–
35	<i>epi</i> -bicyclosesquiphellandrene* ⁶	1623	–	tr	tr	0.2	–	–
36	1- <i>epi</i> -Cubenol* ⁷	1630	1628	2.4	1.9	2.4	2.2	0.3
37	γ -Eudesmol	1634	1632	0.4	0.3	0.5	0.4	0.1
38	<i>epi</i> - α -Cadinol	1643	1638	1.3	0.9	1.4	1.2	0.2
39	Isoaromadendrene epoxide	1656	–	tr	tr	tr	–	–
	Yields (g/kg)			1.4	1.5	1.4	1.4	0.0
	Total listed (%)			90.7	96.4	93.2	93.4	2.8

Notes: ^aCompounds listed in order of elution from an HP-5 MS capillary column. RI^b, retention indices relative to *n*-alkanes C₇–C₂₇ on non-polar column HP-5 MS; RI^c, retention index on DB-5 (30), tr: trace (<0.1%). *Identification tentative. ¹*m/z* (% rel. int.): 100 (119), 96 (105), 95 (161), 47 (91), 29 (81), 25 (93), 23 (41), 22 (204), 20 (120), 20 (92), (79 *m/z*). ²*m/z* (% rel. int.): 100 (119), 76 (121), 58 (93), 45 (204), 44 (105), 38 (91), 27 (41), 22 (79), 21 (77), 20 (107), (119 *m/z*). ³*m/z* (% rel. int.): 100 (91), 98 (69), 92 (77), 81 (105), 43 (94), 38 (120), 36 (56), 33 (204), 30 (121), 25 (161), (99 *m/z*). ⁴*m/z* (% rel. int.): 100 (159), 93 (105), 85 (131), 75 (220), 69 (177), 69 (91), 68 (202), 52 (106), 47 (144), 40 (96), (27 *m/z*). ⁵*m/z* (% rel. int.): 100 (136), 74 (79), 73 (91), 54 (95), 46 (107), 40 (161), 39 (135), 37 (92), 33 (43), 31 (105), (86 *m/z*). ⁶*m/z* (% rel. int.): 100 (119), 46 (204), 30 (162), 27 (105), 26 (121), 19 (189), 15 (134), 12 (95), 12 (81), 11 (133), (91 *m/z*). ⁷*m/z* (% rel. int.): 100 (95), 81 (121), 40 (43), 38 (204), 38 (164), 35 (109), 28 (94), 26 (71), 26 (79), 17 (69), (131 *m/z*).

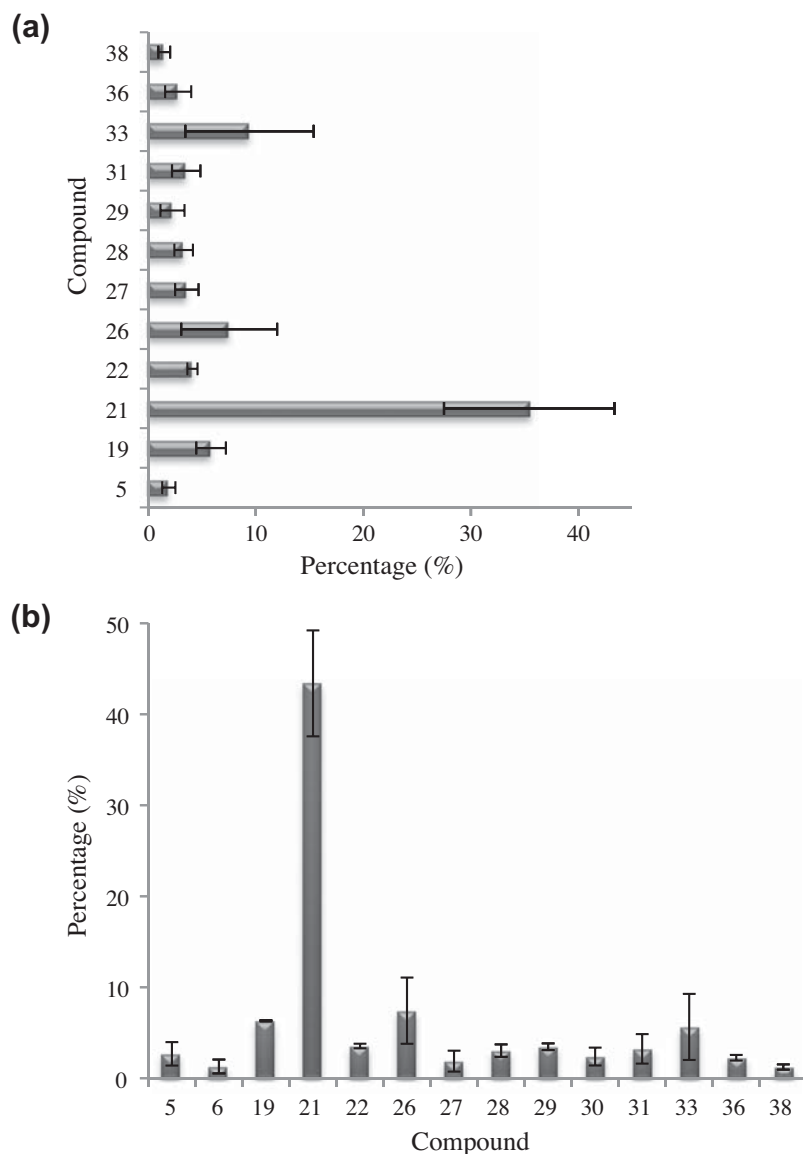


Figure 1. Major compound percentage: (a) within the year 2007 and (b) at March over 3 years (2007–2009).

environmental conditions identical to those of the cultures.

Fumigant toxicity bioassay

A filter paper was treated with an appropriate concentration (1–500 $\mu\text{L/L}$ air) of test essential oil diluted in acetone. The impregnated filter paper was then placed in the bottom cover of a 1-L plastic bottle. The insects, fifty adults with undefined sex per bottle, were exposed for 15 days. Controls received 50 μL acetone. Cumulative mortalities were determined 24, 48, 72, 96 and 120 hours after treatment. All treatments were replicated five times. The long-term activity (remanent effect) of *L. camara* L. leaf essential oils was carried out within a month by introducing new untreated insects every week.

Results and discussion

Essential oil composition

The hydrodistillation of *L. camara* leaves gave an oil in 1.26 ± 0.22 g/kg yield with a maximum in March that equals 1.48 ± 0.05 g/kg over 3 years (2007–2009) based on the dry weight of the plant. The qualitative and quantitative essential oil compositions are presented in Table 1, where compounds are listed in order of their elution on the HP-5 MS column and the mean and standard deviation of the components are given for the year 2007.

The major constituents were β -caryophyllene (46.7–26.3%), caryophyllene oxide (18.8–2.2%) and β -elemene (7.2–3.3%). The composition analysis of the oils revealed that sesquiterpene hydrocarbons predominated (59.2 \pm 9.0%), followed by oxygenated sesquiterpenes

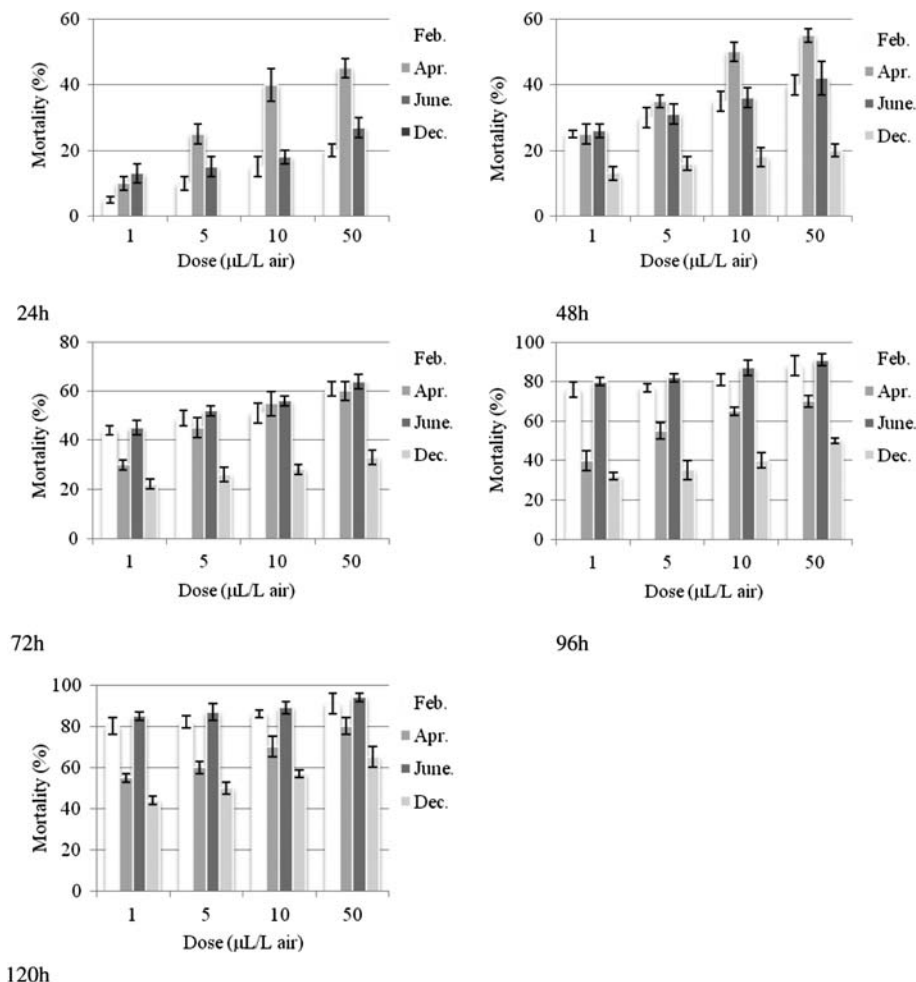


Figure 2. Mortality of *Sitophilus granarius* resulting from 24, 48, 72, 96 and 120 hours of fumigation with different dosages of *Lantana camara* leaf essential oils at 25°C. Means ($n=5$) using fifty adults per replicate.

($11.6\pm 6.4\%$), monoterpenes hydrocarbons ($3.8\pm 0.9\%$) and oxygenated monoterpenes ($1.0\pm 0.5\%$). As shown in Table 1, the range content of some compounds is great, associated with a large standard deviation. This is the case of the most representative components.

The mean and standard deviation of the components at March for the three studied years are also considered. As it can be seen (Table 2), essential oils from March compared with June oil are characterized by a low content in oxygenated sesquiterpenes, among them caryophyllene oxide (3.6% vs. 10.9%). Sesquiterpene hydrocarbon compounds are found in higher amount in March, since α -zingiberene was detected in this type (3.1% vs. 0.2%). For sabinene, a similar result was observed, i.e. a higher amount in March (4.0% vs. 1.4%). Alike results were also observed for β -caryophyllene (47.1% vs. 34.4%). Standard deviation values in Table 2 show that the chemical composition of the yellow–orange Algerian *L. camara* leaf essential oil is relatively stable

because no clear chemical composition change was observed during the three investigated years. We present in Figure 1 the major compound within the year 2007 and its values at March during the three considered years (2007–2009).

Fumigant toxicity

For 24, 48, 76, 96 and 120 hours of fumigation, mortalities at concentrations of 1, 5, 10 and 50 $\mu\text{L/L}$ air for *S. granarius* are shown in Figure 2 for February, April, June and December oils. Concentrations of *L. camara* leaf essential oil of 1, 5, 10 and 50 $\mu\text{L/L}$ air showed 25%, 35%, 50% and 55% kill respectively after 48 hours fumigation for April oil. February, April, June and December oils showed 88%, 70%, 91% and 50% mortality after 120 hours of fumigation. For 72 hours of fumigation, June oil guaranteed 52% kill for only 5 $\mu\text{L/L}$ air. Complete controls can be achieved by treating air with 50 $\mu\text{L/L}$ for 92 hours. However, when developing a new fumigant to meet regulatory requirements,

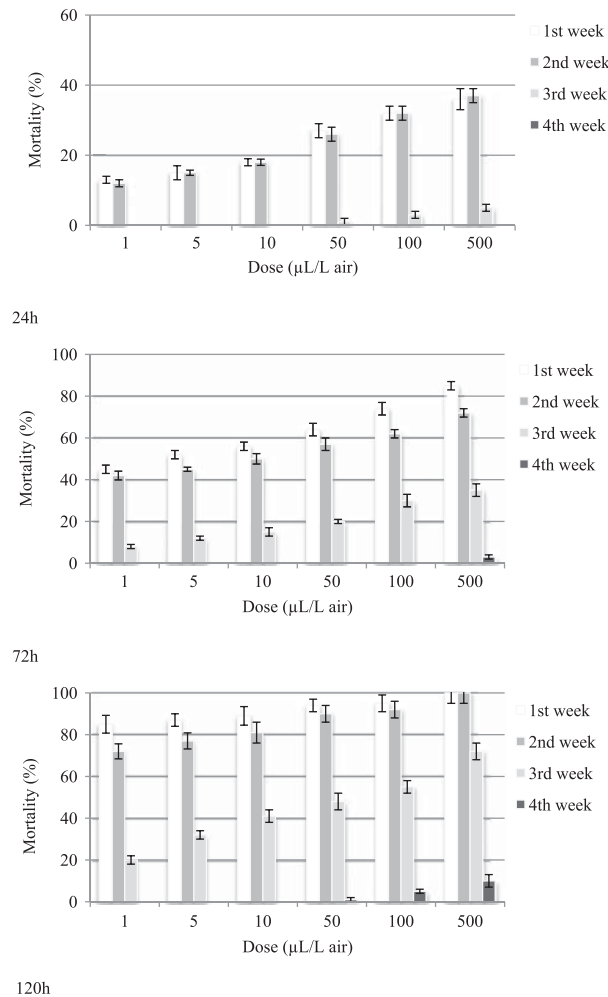


Figure 3. Remanent activity of June *Lantana camara* L. leaf essential oil within 1 month on *Sitophilus granarius* mortality resulting from 24, 72 and 120 hours of fumigation at 25°C. Means ($n=5$) using fifty adults per replicate.

it is necessary to have an understanding of the fumigant toxicity to the target insect pests (29).

The long-term activity of the essential oil was determined for June essential oil. This study was carried out within 1 month by introducing new untreated insects every week. *Sitophilus granarius* mortalities resulting following 1, 2, 3 and 4 weeks of treatment were regrouped in Figure 3. These results confirm that the tested oil was still efficient for 2 weeks, assuring over than 90% of the initial mortality. Again, we record that 500 µL/L assured 100% mortality over the 2 weeks. After 3 weeks, mortalities show a diminution of 50% of the initial record after 120 hours of fumigation. The oil becomes inefficient after the fourth week and cannot ensure more than 10% mortality after 120 hours of fumigation at dose of 500 µL/L.

Lantana camara leaf essential oil shows promise as a material for use as a fumigant. However, field trials

with suitable formulations need to be carried out to further assess the efficacies of essential oils as fumigants.

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