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RESEARCH ARTICLE

Essential oils from three Algerian medicinal plants (*Artemisia campestris*, *Pulicaria arabica*, and *Saccocalyx satureioides*) as new botanical insecticides?

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Abstract

Medicinal and aromatic plants represent an outstanding source of green active ingredients for a broad range of real-world applications. In the present study, we investigated the insecticidal potential of the essential oils obtained from three medicinal and aromatic plants of economic importance in Algeria, *Artemisia campestris*, *Pulicaria arabica*, and *Saccocalyx satureioides*. Gas chromatography coupled with mass spectrometry (GC-MS) was used to study the essential oil chemical compositions. The three essential oils were tested against a mosquito vectoring filariasis and arboviruses, i.e., *Culex quinquefasciatus*, a fly pest acting also as pathogens vector, *Musca domestica*, and an agricultural moth pest, i.e., *Spodoptera littoralis*, using WHO and topical application methods, respectively. The essential oil from *A. campestris*, containing β-pinene (15.2%), α-pinene (11.2%), myrcene (10.3%), germacrene D (9.0%) (*Z*)-β-ocimene (8.1%) and γ-curcumene (6.4%), showed remarkable toxicity against *C. quinquefasciatus* (LC_{50} of 45.8 mg L^{-1}) and moderate effects (LD_{50} of 99.8 μg $adult^{-1}$) against *M. domestica*. Those from *P. arabica* and *S. satureioides*, containing *epi*-α-cadinol (23.9%), δ-cadinene (21.1%), α-cadinol (19.8%) and germacrene D-4-ol (8.4%), and thymol (25.6%), α-terpineol (24.6%), borneol (17.4%) and *p*-cymene (11.4%), respectively, were more active on *S. littoralis* showing LD_{50} values of 68.9 and 61.2 μg $larva^{-1}$, respectively. Based on our results, the essential oil from *A. campestris* may be further considered a candidate ingredient for developing botanical larvicides.

Keywords *Culex quinquefasciatus* · Insect pest · Mosquito vector · *Musca domestica* · Green pesticide · *Spodoptera littoralis*

Introduction

Algeria covers an area of 2,381,741 km^2 and is the first major country in Africa, and one of the Mediterranean countries with a remarkable floristic richness that is directly related to its ecosystem and landscape diversity. The number of taxa of its flora is estimated to about 4000 including 300 endemic taxa (Dobignard and Chatelain 2010, 2011a, b) of which approximately 90% are present in the north of the country. Asteraceae and Lamiaceae represent two important families of medicinal and aromatic plants growing in Algeria, including essential oil–producing species. Among them, the genera *Artemisia* L., *Pulicaria* Gaertner and *Saccocalyx* Coss. & Durand are of economic importance in Algeria due to their important uses as foods and medicines (Miara et al. 2019). The Algerian flora comprises nine species of *Artemisia,* thirteen species of *Pulicaria*, and a species of *Saccocalyx* (Quezel and Santa 1963).

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Artemisia campestris L., commonly called "degoufet," "tgouft," "alala," or "tedjouq," is locally known as a medicinal herb indicated for the treatment of digestive troubles, stomach pain, nausea, pain in menstruation and hypertension (Baba Aissa 1991; Hammiche and Maiza 2006; Baba Aissa 1999; Sari et al. 2012; Boudjelal et al. 2013).

Pulicaria arabica (L.) Cass. has been traditionally used in several countries. It is indigenous to Egypt, locally known as "rara ejub," "abu-'ajn-safra," and "deithouth". It is used in folk medicine to treat ulcers (Provencal 2010). In Morocco, it is known as "hatassa" and used traditionally as anti-tobacco (Mouhajir 2002). In the Iranian folk medicine, *P. arabica* is used as an antidiarrheal agent and against schistosomiasis (Ali 2011). In Tunisia, it is used to treat swelling and boils (Abed et al. 2010). On the other hand, no traditional uses are recorded in Algeria.

Saccocalyx satureioides Coss. & Dur. is known in all North African regions as "azir el-ibel" (Trabut 1935), "zaater" (Biondi et al. 2006), and "zaâtar r'mel" (Benabed 2018), alluding to its resemblance with oregano and thyme. It is traditionally recommended for the treatment of diabetes (Allali et al. 2008).

The essential oil of *A. campestris* has been isolated and investigated for its chemical composition and biological activities by several authors in Algeria (Belhattab et al. 2011; Ghorab et al. 2013; Boutemak et al. 2017; Medila et al. 2017; Bakchiche et al. 2019) and other countries (Neffati et al. 2008; Costa et al. 2009; Judzentienea et al. 2010; Akrout et al. 2003, 2010; Dib et al. 2017). Only two investigations has been provided on the essential oil of *P. arabica* concerning the analysis of its chemical constituents (Mossa et al. 1987; Djermane et al. 2016). The chemical composition of the essential oil of *S. satureioides* as well as its biological properties were described in previous studies (Biondi et al. 2006; Laouer et al. 2006; Bendahou et al. 2008; Bendimerad et al. 2009; Benabed 2018; Kherkhache et al. 2018; Zerroug et al. 2011; Belmekki and Bendimerad 2012; Mohamadi et al. 2015; Benahmed et al. 2016). To the best of our knowledge, the insecticidal properties of the essential oils from these three species have not been explored in deep.

Managing insect pests and vectors through environmentally sustainable approaches represent a major and timely challenge (Benelli and Beier 2017; Stevenson et al. 2017; Chaieb et al. 2018; Isman 2018, 2020; Benelli et al. 2019a; Pavela et al. 2019a; Petrović et al. 2019), to relevant non-target effects of synthetic pesticides on human health and environment (Desneux et al. 2007; Ullah et al. 2019a, b; Varikou et al. 2019), and their efficacy threatened by rapid resistance development in target species (Naqqash et al. 2016; Yavaşoglu et al. 2019; Guz et al. 2020). In this framework, the present research was aimed to investigate the insecticidal activity of *A. campestris*, *P. arabica*, and *S. satureioides* essential oils extracted from plants collected in Algeria. The three essential oils were tested as larvicides against the African cotton leafworm *Spodoptera littoralis* (Boisduval) (Lepidoptera: Noctuidae), as well as against the lymphatic filariasis and arbovirus vector *Culex quinquefasciatus* Say (Diptera: Culicidae), for which effective control tools are urgently needed (Wilke et al. 2020). Note that this species is also under investigation as a potential Zika virus vector, with contrasting evidences reported (Benelli and Romano 2017; van den Hurk et al. 2017). Furthermore, the three essential oils were evaluated as adulticides against the common housefly, *Musca domestica* L. (Diptera: Muscidae), a noxious pest, also able to spread dozens of pathogens (Butler et al. 2010). The chemical composition of the three Algerian essential oils was analyzed through GC-MS analysis, discussing the potential contribution of the essential oil major constituents in terms of insecticidal effectiveness.

Materials and methods

Plant material

Plant material was represented by the aerial parts of *A. campestris* (Asteraceae, collected in Salah Bey (Sétif) region, at the end of August 2016), *P. arabica* (Asteraceae, collected at the University of Mohamed Boudiaf Campus in May 2017) and *S. satureioides* (Lamiaceae, collected in El Houamed (M'sila) region in June 2015). The samples (25–40 plants for each species) were representative of the species and their geographic area of distribution. The voucher specimens were deposited in the herbarium of the Department of Nature Sciences and Life, Faculty of Sciences, M'sila.

Essential oil isolation

The aerial parts of the three species (100 g) were subjected to hydrodistillation for 3 h in a Clevenger-type apparatus. The oil yields, estimated on a dry weight basis (*v*/*w*), were 0.45, 0.40, and 2.8%, for *A. campestris* oil, *P. arabica* and *S. satureioides*, respectively.

Chemical analyses

The three essential oils from *A. campestris*, *P. arabica*, and *S. satureioides* were analyzed by GC-MS using an Agilent 6890N gas chromatograph coupled with a 5973N mass spectrometer. The overall analytical conditions adopted were the same used in the works of Zorzetto et al. (2015) and Maggi et al. (2015). Briefly, an HP-5 capillary column, 30 m in length, 0.25-μm internal diameter, 0.1-μm film thickness, was used as stationary phase whereas helium (99.99%) was the mobile phase with a flow rate of 1 mL/min. Oven was thermostated as follows: 60 °C kept for 5 min, then increased up to 220 °C at 4 °C/min, then raised up to 280 °C at 11 °C/min. Injector and detector temperatures were set to

280 °C. The essential oils were diluted 1:100 in analytical grade *n*-hexane (Sigma-Aldrich, Milan, Italy) and 2 μL injected in split mode (ratio, 1:50). Total ion chromatograms were obtained using the electron impact (EI) mode at 70 eV. The identification followed the procedures already reported by Benelli et al. (2017, 2018a) relying on the interactive combination of temperature-programmed retention indices and mass fragmentations with those stored in commercial libraries (Adams, FFNSC2, NIST17 and Wiley275). Semi-quantitative values were obtained from relative peak areas considering the same response factor for all compounds.

Insect rearing

The insect pest species tested in this study, i.e., *C. quinquefasciatus* larvae, *M. domestica* adults, and *S. littoralis* larvae, were reared following the method recently reported by Benelli et al. (2019b). All species were obtained from established laboratory colonies (> 20 generations) and maintained at 25 ± 1 °C, 70 ± 3% R.H., and 16:8 h (L:D).

Insecticidal activity against *Culex quinquefasciatus*

The insecticidal efficacy of *A. campestris*, *P. arabica*, and *S. satureioides* essential oils diluted in dimethyl sulfoxide (DMSO) on *C. quinquefasciatus* 3rd instar larvae was assessed following WHO (1996) with minor changes by Pavela et al. (2018a). Tested concentrations were 20, 40, 60, 80, 100, 120, 150, 200, 250, 300, 400, 500, and 800 mg L^{-1}; each concentration was replicated 4 times on groups of 25 larvae each. The negative control was distilled water with the same amount of DMSO used for dissolving the tested essential oil. The positive control was pyrethrum extract (50%, Sigma-Aldrich, Czech Republic) tested at 0.02, 0.04, 0.06, 0.08, and 1.00 mg L^{-1}. Larval mortality was recorded after 24 h.

Insecticidal activity against *Musca domestica*

Topical application trials were done to shed light on the acute insecticidal activity of *A. campestris*, *P. arabica*, and *S. satureioides* essential oils on *M. domestica* adult females (3–6 days old). Following Benelli et al. (2019b), 1 μL of acetone (Sigma-Aldrich, Germany) plus the essential oil at the following concentrations, 40, 60, 80, 100, 120, 150, 200, 250, 300, 350, 400, 450, and 500 μg adult^{-1} (each concentration was tested on 4 groups of 20 flies each), was applied using a microelectric applicator to the pronotum of houseflies anesthetized with CO_2. Acetone was the negative control. The positive control was pyrethrum extract (50%, Sigma-Aldrich, Czech Republic) tested at 2, 4, 6, 8 and 10 μg adult^{-1}. Then, *M. domestica* flies were moved to a recovery box (10 × 10 × 12 cm, 26 ± 1 °C 16:8 L:D) for 24 h, and mortality was recorded (Benelli et al. 2019b).

Insecticidal activity against *Spodoptera littoralis*

The toxicity of the *A. campestris*, *P. arabica* and *S. satureioides* essential oils to 3rd instar larvae of *S. littoralis* was evaluated through topical application of the essential oils diluted in acetone. Following Benelli et al. (2019b), the larvae were treated on the dorsum with 1 μL of acetone containing 20, 40, 60, 80, 100, 120, 140, 160, 180, and 200 μg larvae^{-1} of *A. campestris*, *P. arabica*, or *S. satureioides* essential oil. Four replicates ($n = 20$ larvae per replicate) for each concentration were done. Acetone was the negative control. The positive control was pyrethrum extract (50%, Sigma-Aldrich, Czech Republic) tested at 4, 8, 12, 16, and 20 μg larvae^{-1}. Larvae were moved to a recovery box (10 × 10 × 7 cm, with thin holes to avoid fumigation effects, 26 ± 1 °C, 70 ± 3% R.H., and 16:8 L:D) for 24 h, then mortality was noted (Benelli et al. 2019b).

Statistical analysis

In insecticidal assays, if control mortality < 20%, the observed mortality rates were corrected using Abbott's formula (Abbott 1925) and probit analysis was used to estimate the $LD_{50(90)}$ and $LC_{50(90)}$ values, with associated 95% confidence limits (CI_{95}) and chi-square for each tested product (Finney 1971). For the calculation of $LC_{50(90)}$, five suitable concentrations were always selected.

Results and discussion

Essential oil chemical compositions

The chemical compositions of the three essential oils are reported in Table 1, where a total of 94 components, accounting for 88.7–99.7% of the total compositions, are listed. Overall, the three essential oils displayed different chemical profiles, as determined by GC-MS (Fig. 1).

The essential oil from *A. campestris* was characterized by two main chemical classes, namely monoterpene hydrocarbons (58.2%) and sesquiterpene hydrocarbons (21.9%), whereas oxygenated monoterpenes and sesquiterpenes gave a minor contribution (2.2 and 6.3%, respectively). Among monoterpenes, the major constituents were β-pinene (15.2%), α-pinene (11.2%), myrcene (10.3%) and (*Z*)-β-ocimene (8.1%), whereas germacrene D (9.0%) and γ-curcumene (6.4%) were the most representative compounds of sesquiterpenes. *Artemisia campestris* is a polymorphic species, including many subspecies and varieties (Dib and El Alaoui-Faris 2019). Therefore, different essential oil chemotypes have been reported in literature. Abidi et al. (2018) studied an *A. campestris* population growing in Tunisia and found β-pinene (36.4%) and 2-undecanone

Table 1 Chemical composition of the essential oils from three Algerian medicinal plants, *Artemisia campestris*, *Pulicaria arabica*, and *Saccocalyx saturejoides*

No.	Component[a]	RI[b]	RI lit.[c]	*Artemisia campestris* (%)[d]	*Pulicaria arabica* (%)[d]	*Saccocalyx saturejoides* (%)[d]	ID[e]
1	Tricyclene	917	921			0.1 ± 0.0	1,2
2	α-Thujene	921	924	0.1 ± 0.0		0.7 ± 0.1	1,2
3	α-Pinene	926	932	11.2 ± 1.8		2.2 ± 0.5	1,2,3
4	Camphene	940	946	0.1 ± 0.0		3.8 ± 0.7	1,2,3
5	Thuja-2,4(10)-diene	945	953	Tr[f]			1,2
6	Sabinene	966	969	0.8 ± 0.2		0.3 ± 0.1	1,2,3
7	β-Pinene	968	974	15.2 ± 2.9		0.3 ± 0.1	1,2,3
8	3-Octanone	986	979			0.1 ± 0.0	1,2
9	Myrcene	989	988	10.3 ± 2.1		0.8 ± 0.2	1,2,3
10	α-Phellandrene	1003	1002			Tr	1,2,3
11	α-Terpinene	1014	1014	0.1 ± 0.0		0.5 ± 0.1	1,2,3
12	*p*-Cymene	1022	1020	4.3 ± 0.8		11.4 ± 2.1	1,2,3
13	Limonene	1025	1024	4.3 ± 0.9		1.0 ± 0.2	1,2,3
14	(*Z*)-β-Ocimene	1037	1032	8.1 ± 1.6			1,2,3
15	(*E*)-β-Ocimene	1047	1044	1.9 ± 3.9			1,2,3
16	γ-Terpinene	1055	1054	1.2 ± 0.3		3.1 ± 0.6	1,2,3
17	*cis*-Sabinene hydrate	1064	1065			Tr	1,2
18	Terpinolene	1085	1086	0.2 ± 0.0		0.1 ± 0.0	1,2,3
19	*p*-Cymenene	1087	1089			Tr	1,2
20	*trans*-Sabinene hydrate	1096	1098			Tr	1,2
21	Linalool	1101	1095	0.1 ± 0.0		1.1	1,2,3
22	α-Campholenal	1124	1122	0.1 ± 0.0		Tr	1,2
23	*allo*-Ocimene	1129	1128	0.2 ± 0.0			1,2
24	*trans*-Pinocarveol	1133	1135	0.5 ± 0.1		Tr	1,2,3
25	Camphor	1139	1141			0.1 ± 0.0	1,2,3
26	Pinocarvone	1158	1160	0.1 ± 0.0			1,2
27	Borneol	1161	1165	Tr		17.4 ± 3.4	1,2,3
28	*p*-Mentha-1,5-dien-8-ol	1165	1166	0.1 ± 0.0			1,2
29	Terpinen-4-ol	1173	1174	0.2 ± 0.0		1.5 ± 0.3	1,2,3
30	*p*-Cymen-8-ol	1184	1179	0.1 ± 0.0		0.1 ± 0.0	1,2
31	α-Terpineol	1187	1186	0.2 ± 0.0		24.6 ± 4.8	1,2,3
32	Myrtenal	1191	1195	0.2 ± 0.0			1,2,3
33	Myrtenol	1192	1194	0.3 ± 0.1			1,2,3
34	*cis*-Dihydro carvone	1193	1191			0.2 ± 0.0	1,2
35	*trans*-Carveol	1217	1215	Tr			1,2
36	(3*Z*)-Hexenyl 3-methyl butanoate	1238	1232	Tr			1,2
37	Thymol	1295	1289	Tr		25.6 ± 4.9	1,2,3
38	Carvacrol	1303	1298	Tr		2.1 ± 0.4	1,2,3
39	(3*Z*)-Hexenyl tiglate	1326	1325	Tr			1,2
40	δ-Elemene	1331	1335	0.1 ± 0.0			1,2
41	α-Cubebene	1344	1345		Tr		1,2
42	Thymol acetate	1354	1349			Tr	1,2
43	α-Copaene	1368	1374	0.4 ± 0.1	0.1 ± 0.0		1,2
44	β-Bourbonene	1376	1387	0.1 ± 0.0			1,2
45	(*E*)-β-Damascenone	1380	1383		Tr		1,2,3
46	β-Cubebene	1383	1387		Tr		1,2
47	α-Gurjunene	1400	1409		0.2 ± 0.0	0.1 ± 0.0	1,2
48	(*E*)-Caryophyllene	1409	1417	0.5 ± 0.1	0.1 ± 0.0	0.6 ± 0.2	1,2,3
49	β-Copaene	1420	1430	Tr	Tr		1,2
50	*cis*-Muurola-3,5-diene	1438	1448		0.1 ± 0.0		1,2
51	*trans*-Muurola-3,5-diene	1442	1451		0.1 ± 0.0		1,2
52	(*Z*)-β-Farnesene	1439	1440	Tr			1,2
53	α-Humulene	1443	1452	Tr	Tr	0.1 ± 0.0	1,2,3
54	*allo*-Aromadendrene	1451	1458		Tr	0.1 ± 0.0	1,2,3
55	*cis*-Cadina-1(6),4-diene	1454	1461		0.2 ± 0.0		1,2
56	(*E*)-β-Farnesene	1456	1454	0.1 ± 0.0	Tr		1,2,3
57	γ-Muurolene	1470	1478		0.8 ± 0.2		1,2
58	Germacrene D	1472	1484	9.0 ± 1.7	0.1 ± 0.0		1,2
59	*trans*-Cadina-1(6),4-diene	1475	1471		3.2 ± 0.7		1,2
60	γ-Curcumene	1475	1481	6.4 ± 1.3			1,2
61	*ar*-Curcumene	1479	1478	2.4 ± 0.5			1,2
62	*trans*-Muurola-4(14),5-diene	1482	1493		0.5 ± 0.1		1,2
63	Thymol isobutyrate	1484	1480		1.6 ± 0.3		1,2
64	δ-Selinene	1484	1492		1.9 ± 0.4		1,2

Table 1 (continued)

No.	Component[a]	RI[b]	RI lit.[c]	*Artemisia campestris* (%)[d]	*Pulicaria arabica* (%)[d]	*Saccocalyx saturejoides* (%)[d]	ID[e]
65	*epi*-Cubebol	1486	1493		0.4 ± 0.1		1,2
66	Bicyclogermacrene	1487	1500	2.1 ± 0.4		0.2 ± 0.0	1,2
67	α-Zingiberene	1492	1492	0.1 ± 0.0			1,2
68	α-Muurolene	1493	1500	0.1 ± 0.0	3.4 ± 0.7	Tr	1,2
69	γ-Cadinene	1506	1513	0.1 ± 0.0	2.6 ± 0.5	0.1 ± 0.0	1,2
70	β-Curcumene	1508	1514	0.1 ± 0.0			1,2
71	δ-Cadinene	1517	1520	0.5 ± 0.1	21.1 ± 3.8	0.3 ± 0.1	1,2
72	*trans*-Cadina-1,4-diene	1525	1533		0.3 ± 0.0		1,2
73	α-Cadinene	1530	1537		0.6 ± 0.2		1,2
74	α-Calacorene	1535	1544		Tr		1,2
75	(*E*)-Nerolidol	1562	1561	0.1 ± 0.0			1,2,3
76	Germacrene D-4-ol	1567	1574		8.4 ± 1.7		1,2
77	Spathulenol	1567	1577	4.5 ± 0.9		0.3 ± 0.1	1,2
78	Caryophyllene oxide	1571	1583	0.2 ± 0.0			1,2,3
79	*ar*-Turmerol	1576	1582	0.2 ± 0.0			1,2
80	Salvial-4(14)-en-1-one	1583	1594	0.1 ± 0.0			1,2
81	Neryl isovalerate	1585	1582	Tr			1,2
82	Ledol	1591	1602			0.1 ± 0.0	1,2
83	1,10-*di-epi*-Cubenol	1605	1618		0.2 ± 0.0		1,2
84	Geranyl isovalerate	1609	1606	0.3 ± 0.0			1,2
85	1-*epi*-Cubenol	1619	1627		0.6 ± 0.1		1,2
86	*epi*-α-Cadinol	1633	1638	0.1 ± 0.0	23.9 ± 4.2	0.1 ± 0.0	1,2
87	*epi*-α-Muurolol	1633	1640	0.1 ± 0.0		0.1 ± 0.0	1,2
88	α-Muurolol	1639	1644		2.5 ± 0.5		1,2
89	β-Eudesmol	1639	1649	0.9 ± 0.2			1,2
90	α-Cadinol	1646	1652	0.2 ± 0.0	19.8 ± 3.9	0.1 ± 0.0	1,2
91	Shyobunol	1678	1688			0.3 ± 0.0	1,2
92	Oplopanone	1727	1739		Tr		1,2
93	Cyclocolorenone	1745	1759		0.1 ± 0.0		1,2
94	Phytone	1844	1845		Tr		1,2
	Total identified (%)			88.7	93.2	99.7	
	Chemical classes						
	Monoterpene hydrocarbons			58.2		24.4	
	Oxygenated monoterpenes			2.2	1.6	72.9	
	Sesquiterpene hydrocarbons			21.9	35.6	1.3	
	Oxygenated sesquiterpenes			6.3	56.0	0.9	
	Others			0.1	tr	0.1	

[a] Components are listed according to their elution from a HP-5MS column

[b] Linear retention index (RI) experimentally calculated using the Van den Dool and Kratz formula (1963) using a mixture of *n*-alkanes (C_8-C_{24})

[c] Literature RI taken from NIST 17 or ADAMS libraries

[d] Relative peak area percentage as mean of three measurements ± SD

[e] Identification: *RI*, correspondence of calculated value with those stored in NIST 17 or ADAMS libraries; *MS*, similarity of mass fragmentation with those recorded in NIST 17, WILEY 275, FFNSC2, and ADAMS libraries; *Std*, comparison with available analytical standard (Sigma-Aldrich, Milan, Italy)

[f] *Tr*, traces, % < 0.1

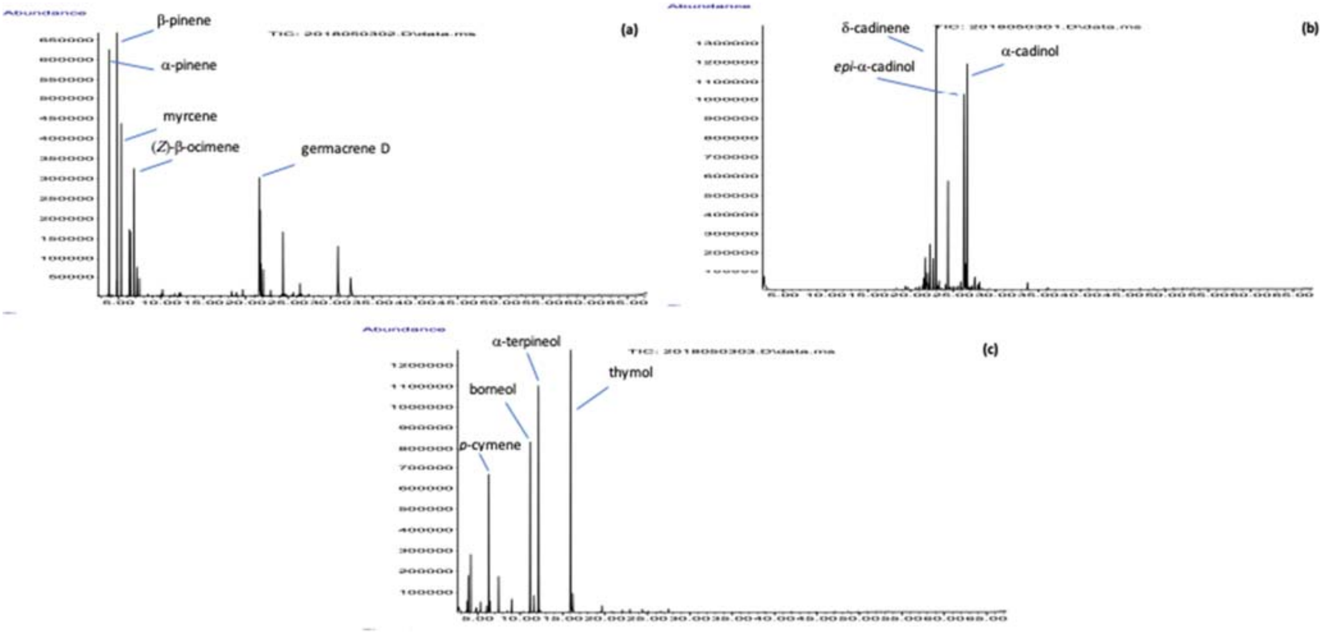

Fig. 1 TIC chromatograms of the three essential oils from *Artemisia campestris* (**a**), *Pulicaria arabica* (**b**), and *Saccocalyx saturejoides* (**c**)

(14.7%) as the major volatile constituents. Other Tunisian populations of *A. campestris* were analyzed by Younsi et al. (2017) who detected germacrene D (16.38%), β-pinene (16.33%) and limonene (9.17%) as the main essential oil compounds. Dib et al. (2017) investigated the essential oil composition of a population growing in Morocco finding spathulenol (10.2%) as the main component. Judzentiene and Budiene (2014) studied natural populations growing in Lithuania and found germacrene D (9.8–31.2%) as the major volatile compound in all the samples analyzed. Other noteworthy compounds detected in these populations were α-pinene, β-pinene, caryophyllene oxide, γ-curcumene and β-selinene. Boukhalkhal et al. (2018) identified a new chemotype containing the polyacetylene capillene in a population belonging to the subsp. *campestris* growing in Algeria. On the other hand, Houicher et al. (2016) reported a similar chemical profile for a natural population growing in Algeria, with α-pinene (18.7%), β-pinene (16.8%), β-myrcene (17.3%) and germacrene D (10.3%) as the major essential oil constituents.

The essential oil from *P. arabica* was dominated by oxygenated sesquiterpenes (56.0%) and sesquiterpene hydrocarbons (35.6%), with *epi*-α-cadinol (23.9%), δ-cadinene (21.1%), α-cadinol (19.8%), and germacrene D-4-ol (8.4%) as the most abundant compounds. The present work represents the second investigation about the chemical composition of the *P. arabica* essential oil. Previously, Mossa et al. (1987) investigated the essential chemical composition of natural populations growing in Saudi Arabia finding sesquiterpene hydrocarbons as the main volatile fraction. However, results were not detailed since many compounds have not been identified. Therefore, a comparative evaluation between the two studies cannot be done. Within the genus *Pulicaria*, *P. undulata* (L.) C.A.Mey was the subject of several investigations for essential oil composition. For instance, Mustafa et al. (2018) found carvacrol (46.5%), xanthoxylin (18.1%), and carvotanacetone (8.7%) as the most abundant volatile compounds in a population growing wild in Egypt. Boumaraf et al. (2016) analyzed the essential oil of *P. undulata* growing in Algeria reporting carvotanacetone (14.8%), δ-cadinene (8.2%), α-cadinol (4.7%), and thujanol (4.7%) as the main constituents. Ravandeh et al. (2011) studied the essential oil composition of an Iranian population, finding terpinen-4-ol (20.1%), *cis*-calamenene (13.37%), junipene (8.66%), *cis*-sabinene hydrate (8.29%), and γ-terpinene (7.00%) as the main constituents. Thus, the *Pulicaria* essential oils seem to be quite variable depending on genetics and geographic factors (see also Znini et al. 2013).

The essential oil from *S. satureioides* was dominated by oxygenated monoterpenes (72.9%) followed by monoterpene hydrocarbons (24.4%), whereas the other groups gave scant contributions. The major components were thymol (25.6%), α-terpineol (24.6%), borneol (17.4%) and *p*-cymene (11.4%). Several research groups analyzed the essential oil chemical profile of *S. satureioides* populations growing in Algeria. Bendahou et al. (2008), Biondi et al. (2006) and Laouer et al. (2006) found an essential oil chemical profile qualitatively similar to that detected by us, with borneol (27.4%), α-terpineol (17.2%) and thymol (18.8%); α-terpineol (32.7%), thymol (22.8%), borneol (11.6%) and carvacrol (6.9%); and α-terpineol (35.9%), thymol (15.6%) and borneol (12.4%) as the main constituents, respectively. Based on these studies, it seems that the essential oil of *S. satureioides* growing in

Algeria is characterized by the thymol/α-terpineol/borneol chemotype, with slight differences in percentages depending on the harvesting times, plant processing and analytical conditions adopted for analyses.

Insecticidal activity

The essential oils extracted from the *A. campestris*, *P. arabica* and *S. satureioides* were toxic to the three insect species tested in this study (Table 2). As a general trend, toxicity was significantly lower than that characterizing the positive control, pyrethrum extract (Table 2). Concerning *C. quinquefasciatus* 3rd instar larvae, the most effective essential oil was the *A. campestris* one, showing a LC_{50} value of 45.8 mg L^{-1}, significantly lower if compared to the LC_{50} values achieved by the essential oils of *S. satureioides* and *P. arabica*, which were 62.8 and 230.3 mg L^{-1}, respectively, with no overlapping CI_{95}. Both *A. campestris* and *S. satureioides* had LC_{90} values lower than 100 mg L^{-1}, i.e., 84.2 and 94.4 mg L^{-1} indicating promising insecticidal activity (Table 2), which is generally considered suitable for the development of botanical larvicides (Pavela 2015).

The *A. campestris* essential oil was toxic also against *M. domestica* adults, showing a LD_{50} value of 99.8 μg $adult^{-1}$. However, this LD_{50} value did not significantly differ (with overlapping CI_{95}) from the LD_{50} estimated for *S. satureioides* essential oil, which was 113.9 μg $adult^{-1}$. On the other hand, the LD_{50} calculated for the *P. arabica* essential oil (193.3 μg $adult^{-1}$) was significantly higher if compared to the two abovementioned ones. Overall, to suppress 90% of the housefly population with the most effective essential oil, *A. campestris*, a LD_{90} of 258.7 μg $adult^{-1}$ is needed (Table 2).

The essential oil from *A. campestris* was scarcely toxic to *S. littoralis* 3rd instar larvae, with an $LD_{50} > 200$ μg $larva^{-1}$, while *S. satureioides* and *P. arabica* essential oils achieved LD_{50} values of 61.2 and 68.9 μg $larva^{-1}$, respectively, with overlapping CI_{95}. Their LD_{90} did not differ with each other, being 150.2 and 131.3 μg $larva^{-1}$, respectively, with overlapping CI_{95}. All essential oils also showed significantly lower efficacy if compared to the pyrethrum extract tested by us as positive control (Table 2).

Although the efficacy of the essential oils we tested was average compared to some other essential oils (Palacios et al. 2009a, b), it can be assumed that even sub-lethal doses can have fatal outcomes, for example, against flies, as previously demonstrated for the essential oils from *Thymus vulgaris* L. containing thymol as its major substance. Sub-lethal doses of thyme and *Carlina acaulis* L. essential oils caused a significant reduction in the longevity and fecundity of *M. domestica*, thyme oil exposure also led to lower weight of the pupae, high mortality during metamorphosis and very low total F_1 natality when the parental females had been exposed (Pavela 2007; Pavela et al. 2020).

Table 2 Insecticidal efficacy of the essential oils (EOs) from three Algerian medicinal plants, *Artemisia campestris*, *Pulicaria arabica* and *Saccocalyx saturejoides*, on larvae of *Culex quinquefasciatus* and *Spodoptera littoralis*, and adults of *Musca domestica*

Insect species	Unit	LC_{50}/LD_{50} ± SE	CI_{95}	LC_{90}/LD_{90} ± SE	CI_{95}	χ^2	*p*-value
Artemisia campestris EO							
Culex quinquefasciatus 3rd instar larva	mg L^{-1}	45.8 ± 1.8	46.5–49.4	84.2 ± 5.8	75.9–82.9	6.849	0.144 n.s.
Musca domestica adult female	μg $adult^{-1}$	99.8 ± 5.6	89.5–111.3	258.7 ± 31.2	212.3–340.8	1.071	0.898 n.s.
Spodoptera littoralis 3rd instar larva	μg $larva^{-1}$	> 200					
Pulicaria arabica EO							
Culex quinquefasciatus 3rd instar larva	mg L^{-1}	230.3 ± 6.8	228.6–245.5	841.3 ± 87.6	729.9–953.9	4.077	0.133 n.s.
Musca domestica adult female	μg $adult^{-1}$	193.3 ± 22.3	174.5–231.8	523.4 ± 17.5	498.3–597.8	0.801	0.671 n.s.
Spodoptera littoralis 3rd instar larva	μg $larva^{-1}$	68.9 ± 4.9	59.3–72.9	131.3 ± 10.2	125.8–148.6	0.678	0.715 n.s.
Saccocalyx saturejoides EO							
Culex quinquefasciatus 3rd instar larva	mg L^{-1}	62.8 ± 1.9	59.2–66.9	94.4 ± 5.4	86.1–99.1	1.882	0.391 n.s.
Musca domestica adult female	μg $adult^{-1}$	113.9 ± 6.1	102.6–126.7	260.4 ± 31.4	256.7–325.8	1.191	0.755 n.s.
Spodoptera littoralis 3rd instar larva	μg $larva^{-1}$	61.2 ± 9.2	48.9–82.3	150.2 ± 12.3	142.2–172.9	0.225	0.882 n.s.
Positive control: pyrethrum extract							
Culex quinquefasciatus 3rd instar larva	mg L^{-1}	0.071 ± 0.001	0.068–0.73	0.095 ± 0.003	0.090–0.102	1.889	0.929 n.s.
Musca domestica adult female	μg $adult^{-1}$	3.98 ± 0.25	3.25–4.56	9.87 ± 0.85	8.79–11.56	2.145	0.845 n.s.
Spodoptera littoralis 3rd instar larva	μg $larva^{-1}$	5.83 ± 1.15	4.95–6.78	15.18 ± 1.12	12.35–17.12	1.285	0.529 n.s.

LC/LD_{50} = lethal concentration/lethal dose killing 50% of the exposed insect population

LC/LD_{50} = lethal concentration/lethal dose killing 90% of the exposed insect population

CI_{95} = 95% confidence interval

n.s. = not significant (*d.f.* = 4, $p > 0.05$)

This phenomenon has also been observed for other insect species, where it has been found that application of sub-lethal doses or concentrations may have a significant negative effect on larval development and on reproductive parameters in adults (Pavela 2013; Benelli et al. 2018b). However, we are aware that further experiments will be needed to confirm this hypothesis.

Overall, the *A. campestris* essential oil appeared to be the most active on the lymphatic filariasis vector, *C. quinquefasciatus*. The chemical analysis of its essential oil put in evidence a pool of major constituents, namely β-pinene (15.2%), α-pinene (11.2%), myrcene (10.3%), germacrene D (9.0%), (*Z*)-β-ocimene (8.1%) and γ-curcumene (6.4%). The larvicidal effect can therefore be the result of the complex interaction between these compounds. The two pinene isomers (α- and β-) are endowed with larvicidal effects against several mosquito larvae (Govindarajan et al. 2016). For instance, α-pinene showed a LC_{50} values of 15.4 and 47–49 ppm on *Aedes aegypti* L. and *C. pipiens*, respectively; β-pinene displayed LC_{50} values of 12.1, 23.2, and 32.2 ppm on *Ae. aegypti*, *Anopheles stephensi* Liston, and *C. quinquefasciatus*, respectively; germacrene D was toxic to *An. anthropophagus* larvae with LC_{50} of 49.8 ppm; (*Z*)-β-ocimene was toxic to *An. stephensi*, *Ae. aegypti*, and *C. quinquefasciatus* larvae showing LC_{50} of 41.9, 46.3 and 50.6 ppm, respectively (Maggi and Benelli 2018). In particular, the presence of exocyclic double bonds in some of their structures may contribute to inducing toxicity in insects (Benelli et al. 2017). Taken together, these mixtures may also have a multi-target action, interacting with GABA, AChE and octopaminergic systems (Jankowska et al. 2017, 2019).

On the other hand, *S. satureioides* and *P. arabica* essential oils achieved noteworthy toxicity on *S. littoralis*. The two essential oils were characterized by thymol (25.6%), α-terpineol (24.6%), borneol (17.4%) and *p*-cymene (11.4%), and *epi*-α-cadinol (23.9%), δ-cadinene (21.1%), α-cadinol (19.8%), and germacrene D-4-ol (8.4%), respectively. The toxicity of thymol-rich essential oil from *S. satureioides* and thymol itself in *S. littoralis* has been reported in several papers (Pavela 2010; Pavela et al. 2019c). One of the main mechanisms of action of thymol is through modulation of GABAA receptors (Priestley et al. 2003). It has also been reported that the effect of thymol may be enhanced by the presence of *p*-cymene (Burt 2004). However, it is surprising that this kind of oil did not produce relevant toxicity on *C. quinquefasciatus* and *M. domestica*, as previously seen for other oils with similar chemical profile or for the pure compound itself (Benelli et al. 2017; Pavela et al. 2018b). Thus, we can assume that the other constituents may antagonize the mosquitocidal effects of thymol. Concerning the oxygenated sesquiterpenes, the main essential oil constituents in *P. arabica*, no reports on their insecticidal effects have been previously provided.

The essential oils tested in this study were extracted from plants growing in arid areas. Therefore, they may have a relevant potential as novel crops in climatically dry areas in the future. *Saccocalyx satureioides*, which had a high yield of essential oil (2.8%), appears to be particularly promising as source of novel green insecticides. Further research on this plant should focus on new technologies to boost its effectiveness and stability in the field (Pavoni et al. 2019), as well as the possibility of increasing the yield of essential oils, which has proved to be very important for the cultivation of aromatic plants (Pavela et al. 2016, 2018c).

Conclusions

The exploration of the Algerian flora in the search of bioactive plant-borne essential oils led us to identify *A. campestris* as a potential ingredient for botanical insecticides. From a real-world point of view, the most interesting use of the *A. campestris* essential oil is as mosquito larvicide, as the LC_{50} fits the criteria by Pavela (2015), being below 100 mg L^{-1}. Future research efforts are needed about evaluating the synergistic and antagonistic blends of the *A. campestris* essential oil in selected blends of botanical insecticides for biocontrol purposes (Benelli et al. 2017), as well as to shed light on the possible modes of action of this essential oil (Jankowska et al. 2017) and to boost its stability and bioactivity through nanoemulsion and nanoencapsulation techniques or the employ of green coated nanoparticles (Benelli et al. 2018c; Pavela et al. 2019b).

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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