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## ORIGINAL ARTICLE

# Characterization of leaves and flowers volatile constituents of *Lantana camara* growing in central region of Saudi Arabia

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## KEYWORDS

Verbenaceae;  
Essential oils;  
*cis*-3-Hexen-1-ol;  
1-Octen-3-ol;  
 $\beta$ -Caryophyllene;  
*Lantana camara*

**Abstract** The chemical components of essential oils derived from leaves and flowers of *Lantana camara* growing in Saudi Arabia are analyzed for the first time using gas chromatography techniques (GC–MS, GC–FID, Co-GC, LRI determination, and database and literature searches) on two different stationary phase columns (polar and nonpolar). This analysis led to the identification of total 163 compounds from leaves and flowers oils. 134 compounds were identified in the oil obtained from leaves of *L. camara*, whereas 127 compounds were identified in the oil obtained from flowers; these compounds account for 96.3% and 95.3% of the oil composition, respectively. The major components in the oil from leaves were *cis*-3-hexen-1-ol (11.3%), 1-octen-3-ol (8.7%), spathulenol (8.6%), caryophyllene oxide (7.5%) and 1-hexanol (5.8%). In contrast, the major compounds in the flowers oil were caryophyllene oxide (10.6%),  $\beta$ -caryophyllene (9.7%), spathulenol (8.6%),  $\gamma$ -cadinene (5.6%) and *trans*- $\beta$ -farnesene (5.0%). To the best of our knowledge, *cis*-3-hexen-1-ol and 1-octen-3-ol that were identified as major components in this study have not been reported earlier from *Lantana* oils.

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## 1. Introduction

*Lantana* is a genus of both herbaceous plants and shrubs containing about 150 species and belongs to the family Verbenaceae (Ghisalberti, 2000). *Lantana camara* is an evergreen climbing aromatic shrub of the genus *Lantana* and is considered to be one of the most important medicinal plants of the world (Sharma et al., 2000; Srivastava et al., 2005). It can grow up to 2–4 m in height under normal conditions but has the ability to climb up to 15 m in height with the support of surrounding vegetation (Day et al., 2003). *L. camara* is native to tropical regions of America and Africa, but now, it has been introduced as an ornamental plant in most countries worldwide

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including Saudi Arabia and has been completely naturalized in most tropical and subtropical parts of the world as it can easily grow and survive in variety of agro-climatic conditions (Sharma, 1981).

*L. camara* have been widely used in traditional medicine for the treatment of malaria, ulcers, cancer, high blood pressure, tetanus, tumors, eczema, cuts, catarrhal infections, atoxy of abdominal viscera, chicken pox, measles, rheumatism, asthma and fevers (Day et al., 2003; Ghisalberti, 2000; Lenika et al., 2005; Sathish et al., 2011). It is an excellent provenance for several classes of bioactive natural products including triterpenoids, flavonoids, steroids, iridoide glycosides, oligosaccharides, phenylpropanoid glycosides, and naphthoquinones (Begum et al., 2014; Sharma et al., 2007; Sousa et al., 2012). Varieties of lead phytomolecules such as oleanolic acid, ursolic acid, lantanoside, linarioside, camarinic acid, verbascoside, umuhengerin and phytol have been isolated from *L. camara* and their various biological activities such as hepatoprotective, leishmanicidal, anticancer, antibacterial, antioxidant, antimycobacterial, nematocidal, and antiulcer have been reported (Begum et al., 2014, 2008, 1995; Day et al., 2003; Herbert et al., 1991; Sathish et al., 2011; Qamar et al., 2005). Roots of *L. camara* have been described to be a rich and an inexpensive source of putative biologically active compound "oleanolic acid" for which some optimized and economical isolation procedures have been described and the isolation process has been patented (Banik and Pandey, 2008; Misra et al., 1997; Srivastava et al., 2005; Verma et al., 2013). Moreover, *L. camara* has been proven to be one of the most easily available and cheap materials for the isolation of industrial essential oils famously known as *Lantana* oils (Randrianalijaona et al., 2005; Weyerstahl et al., 1999). Essential oils isolated from various parts of *L. camara* from different regions of the world have previously been studied (Filho et al., 2012; Kasali et al., 2004; Khan et al., 2002; Love et al., 2009; Ngassoum et al., 1999; Padalia et al., 2010; Sefidkon, 2002; Sundufu and Shoushan, 2004) and shown to possess various biological activities such as anti-inflammatory (Benites et al., 2009), antibacterial (Tesch et al., 2011), antioxidant (Sousa et al., 2013), insecticidal (Zoubiri and Baaliouamer, 2012b), allelopathic (Verdeguer et al., 2009) and larvicidal (Dua et al., 2010). Owing to the rapid propagation, invasive nature and abundant availability of *L. camara*, extensive research work in several parts of the world are going on in order to make this plant more useful for industrial applications (Passos et al., 2012; Patel, 2011; Sousa et al., 2013). In continuation of our research interest in exploring various medicinal and aromatic plants grown in diverse agro-climatic conditions (Al-Mazroa et al., 2015; Al-Otaibi et al., 2014; Khan et al., 2014, 2012, 2006), we have previously reported essential oil compositions of *L. camara* from India and developed an economical process for the isolation of hepatoprotective agent "oleanolic acid" from the root of *L. camara* (Khan et al., 2003; Srivastava et al., 2005). Herein, we are reporting detail chemical characterization of volatile constituents of leaves and flowers essential oils of *L. camara* grown in Saudi Arabia using GC-FID and GC-MS analyses as well as linear retention indices (LRI) measurements performed on both polar and nonpolar columns. To the best of our knowledge, this is the first report on phytochemical investigation of *L. camara* growing in Saudi Arabian agro-climatic conditions.

## 2. Experimental

### 2.1. Plant material

The whole plant of *L. camara* was procured from Riyadh, central part of Saudi Arabia during the flowering stage in the month of April 2011. The identification of the plant species was confirmed by a botanical taxonomist (Dr. Jacob Thomas Pandalayil) from the Herbarium Division, College of Science, King Saud University, Riyadh, KSA. The voucher specimen (No. KSUHJK-301) of the plant material is maintained in our laboratory.

### 2.2. Isolation of essential oils

The leaves and flowers from freshly collected *L. camara* plant material were separated and sliced into small pieces. The sliced fresh leaves (290.0 g) and flowers (475.0 g) were separately subjected to hydro-distillation for 3 h using a Clevenger-type apparatus according to the European Pharmacopoeia method (European Pharmacopoeia, 1996) to give light-orange color oils. The oils obtained after the hydro-distillation were dried over anhydrous sodium sulfate and stored at 4 °C until further use. The yield of the volatile oils derived from the leaves and flowers was 0.06% and 0.08% (w/w), respectively, on the fresh weight basis.

### 2.3. Chemicals

Analytical-grade acetone (Sigma-Aldrich, Germany) was used for the dilution of oil samples. Pure volatile compounds such as linalool, nonanal, limonene, terpinene-4-ol, eugenol,  $\alpha$ -bisabolol, and  $\alpha$ -terpinolene were available in our laboratory and used for co-injection analysis.

### 2.4. GC-FID and GC-MS analyses

The essential oils were analyzed using a GC-MS and GC-FID equipped with two columns, one of which was polar (DB-Wax), and the other was nonpolar (HP-5MS). GC-MS was performed on an Agilent single-quadrupole mass spectrometer with an inert mass selective detector (MSD-5975C detector, Agilent Technologies, USA) coupled directly to an Agilent 7890A gas chromatograph which was equipped with a split-splitless injector, a quickswap assembly, an Agilent model 7693 autosampler and a HP-5MS fused silica capillary column (5% phenyl 95% dimethylpolysiloxane, 30 m  $\times$  0.25 mm i.d., film thickness 0.25  $\mu$ m, Agilent Technologies, USA). Supplementary analyses were performed on a DB-Wax fused silica capillary column (polyethylene glycol, 30 m  $\times$  0.25 mm i.d., film thickness 0.25  $\mu$ m, Agilent Technologies, USA). The HP-5MS column was operated using an injector temperature of 250 °C and the following oven temperature profile: an isothermal hold at 50 °C for 4 min, followed by a ramp of 4 °C/min to 220 °C, an isothermal hold for 2 min, a second ramp to 280 °C at 20 °C/min and finally an isothermal hold for 15 min. Conversely, the DB-Wax column was operated using an injector temperature of 250 °C and the following oven temperature profile: an isothermal hold at 40 °C for 4 min, followed by a ramp of 4 °C/min to 220 °C and an isothermal hold for 10 min.

Approximately 0.2  $\mu$ l of each sample diluted in acetone (5% solution in acetone) was injected using the split injection mode; the split flow ratio was 10:1. The helium carrier gas was flowed at 1 ml/min. The GC-TIC profiles and mass spectra were obtained using the ChemStation data analysis software, version E-02.00.493 (Agilent). All mass spectra were acquired in the EI mode (scan range of  $m/z$  45–600 and ionization energy of 70 eV). The temperatures of the electronic-impact ion source and the MS quadrupole were 230 °C and 150 °C, respectively. The MSD transfer line was maintained at 280 °C for both polar and nonpolar analyses. The GC analysis was performed on an Agilent GC-7890A dual-channel gas chromatograph (Agilent Technologies, USA) equipped with

**Table 1** Composition of essential oils derived from leaves and flowers of *Lantana camara* from the central region of Saudi Arabia.

Sl. No.	Compound*	LRI <sup>a</sup>	LRI <sup>P</sup>	LCL (%)	LCF (%)
1	2,2-Diethoxypropane	777	–	1.0	t
2	Hexanal	800	1080	0.1	–
3	<i>trans</i> -3-Hexen-1-ol	849	1367	0.3	–
4	<i>trans</i> -2-Hexenal	850	1216	0.3	–
<b>5</b>	<b><i>cis</i>-3-Hexen-1-ol</b>	<b>852</b>	<b>1388</b>	<b>11.3</b>	–
6	2-Methyl-butanoic acid	856	1662	–	0.1
7	<i>trans</i> -2-Hexen-1-ol	859	1410	0.1	–
8	<i>cis</i> -2-Hexen-1-ol	863	–	0.6	–
<b>9</b>	<b>1-Hexanol</b>	<b>865</b>	<b>1357</b>	<b>5.8</b>	–
10	1,3,5,7-Cyclooctatetraene	890	–	0.2	0.3
11	<i>n</i> -Nonane	900	900	0.1	–
12	(2 <i>E</i> )-Heptenal	954	–	0.1	0.1
13	Benzaldehyde	960	1512	–	t
14	Verbenone	969	1122	0.1	–
15	Sabinene	973	1121	0.1	–
<b>16</b>	<b>1-Octen-3-ol</b>	<b>978</b>	<b>1454</b>	<b>8.7</b>	<b>1.8</b>
17	3-Octanone	987	1255	0.1	t
18	6-Methyl-5-hepten-2-ol	992	1467	0.1	–
19	3-Octanol	994	1397	0.4	0.1
20	<i>p</i> -Cymene	1025	1268	–	t
21	Limonene	1029	1196	0.1	0.1
22	Benzyl alcohol	1033	1881	0.2	–
23	<i>trans</i> - $\beta$ -Ocimene	1047	–	0.1	0.1
24	<i>trans</i> -2-Octen-1-ol	1063	1611	t	–
25	<i>n</i> -Octanol	1070	1556	–	0.1
26	<i>cis</i> -Linalool oxide	1073	1447	0.1	0.1
27	$\alpha$ -Terpinolene	1088	–	0.1	–
28	<i>trans</i> -Sabinene hydrate	1095	1554	0.1	–
29	Linalool	1099	1550	3.0	0.3
30	Nonanal	1104	1394	–	0.1
31	<i>cis</i> -Thujone	1109	1419	0.2	–
32	<i>cis</i> - <i>p</i> -Menth-2-en-1-ol	1122	1614	0.1	t
33	<i>trans</i> - <i>p</i> -Menth-2-en-1-ol	1137	1585	0.1	t
34	<i>cis</i> -Sabinol	1140	–	0.5	0.1
35	<i>cis</i> -Verbenol	1142	1661	0.2	t
36	<i>trans</i> -Verbenol	1146	1685	1.1	0.2
37	<i>iso</i> -Borneol	1157	1669	0.2	–
38	Pinocarvone	1165	1571	0.1	t
39	Borneol	1167	1707	0.4	0.1
40	Lavandulol	1170	–	0.1	t
41	1-Nonanol	1174	–	0.1	–
42	Terpinen-4-ol	1179	1606	0.2	0.1
43	<i>p</i> -Cymene-8-ol	1186	1853	0.1	0.1
44	$\alpha$ -Terpineol	1191	1701	0.2	0.1
45	Myrtenol	1198	1799	0.1	t
46	<i>cis</i> -Piperitol	1203	1712	0.1	0.1
47	<i>n</i> -Decanal	1208	1495	t	–
48	Verbenone	1211	–	0.5	0.2
49	Linalyl formate	1215	1577	0.1	–
50	<i>trans</i> -Carveol	1220	1840	0.1	t
51	Cuminaldehyde	1242	1785	t	–
52	Piperitone	1254	–	–	t
53	<i>n</i> -Decanol	1271	1756	t	0.1
54	<i>n</i> -Tridecane	1299	1300	–	0.1
55	<i>trans</i> -Pinocarvyl acetate	1302	1653	0.1	0.2
56	(2 <i>E</i> ,4 <i>E</i> )-Decadienal	1321	1810	–	0.1
57	Myrtenyl acetate	1325	1693	0.1	–
58	$\alpha$ -Terpinyl acetate	1349	–	0.1	–
59	$\alpha$ -Cubebene	1353	1459	0.1	0.1
60	Eugenol	1359	–	0.2	–
61	<i>n</i> -Decanoic acid	1372	2274	0.1	0.1
62	$\alpha$ -Copaene	1380	1493	0.2	1.7
63	$\beta$ -Bourbonene	1390	1524	–	t

(continued on next page)

**Table 1** (continued)

Sl. No.	Compound*	LRI <sup>a</sup>	LRI <sup>P</sup>	LCL (%)	LCF (%)
64	$\beta$ -Cubebene	1394	1540	0.2	0.6
65	$\beta$ -Elemene	1399	1590	0.1	0.1
66	<i>n</i> -Tetradecane	1401	1400	0.1	0.1
67	$\alpha$ -Cedrene	1412	1587	0.1	0.1
68	<i>cis</i> - $\alpha$ -Bergamotene	1415	1559	–	t
<b>69</b>	<b><math>\beta</math>-Caryophyllene</b>	<b>1425</b>	<b>1599</b>	<b>3.1</b>	<b>9.7</b>
70	$\beta$ -Copaene	1435	–	0.2	0.7
71	<i>trans</i> - $\alpha$ -Bergamotene	1438	1578	–	t
72	<i>cis</i> - $\beta$ -Farnesene	1446	1655	–	0.1
<b>73</b>	<b><i>trans</i>-<math>\beta</math>-Farnesene</b>	<b>1458</b>	<b>1667</b>	<b>0.7</b>	<b>5.0</b>
74	$\alpha$ -Humulene	1460	1673	–	1.0
75	<i>dehydro</i> -Aromadendrene	1465	1981	–	0.1
76	<i>allo</i> -Aromadendrene	1467	1649	0.3	0.7
77	(+)- <i>epi</i> -Bicyclosesquiphellandrene	1473	1594	0.4	0.1
78	<i>trans</i> -Cadina-1(6),4-diene	1479	2167	0.1	–
79	$\gamma$ -Muurole	1481	1691	0.3	0.8
80	Germacrene-D	–	1712	t	–
81	$\alpha$ -Curcumene	1486	1775	0.4	1.7
82	<i>trans</i> - $\beta$ -Ionone	1489	1942	0.9	0.1
83	Calamenene-10,11-epoxide	1495	1890	0.1	–
84	$\alpha$ -Zingiberene	1496	1718	–	0.1
85	<i>epi</i> -Cubebol	1500	1895	0.8	2.0
86	Bicyclogermacrene	1503	1737	0.1	–
87	$\alpha$ -Muurole	1505	–	0.3	0.9
88	$\alpha$ -Cuprenene	1508	2055	0.1	0.1
89	$\beta$ -Bisabolene	1511	1729	0.7	2.6
90	$\beta$ -Curcumene	1516	1743	0.1	–
<b>91</b>	<b><math>\gamma</math>-Cadinene</b>	<b>1521</b>	<b>1761</b>	<b>3.6</b>	<b>5.6</b>
92	$\beta$ -Sesquiphellandrene	1525	1768	0.1	–
93	$\delta$ -Cadinene	1528	1837	0.9	0.7
94	<i>trans</i> -Cadina-1(2),4-diene	1538	1924	0.3	0.2
95	<i>trans</i> - $\alpha$ -Bisabolene	1540	–	0.3	–
96	$\alpha$ -Cadinene	1543	1767	–	0.1
97	$\alpha$ -Calacorene	1547	1920	0.5	0.8
98	<i>cis</i> -Muurole-5-en-4- $\beta$ -ol	1554	2029	–	0.5
99	Germacrene-B	1556	1823	–	0.2
100	Occidentalol	1557	2236	0.6	–
101	<i>cis</i> -Muurole-5-en-4- $\alpha$ -ol	1560	2092	0.2	0.6
102	<i>trans</i> -Nerolidol	1565	2043	0.3	0.5
103	$\beta$ -Calacorene	1569	1963	–	0.2
104	Dodecanoic acid	1571	2489	0.3	–
105	Acora-3,5-dien-11-ol	1576	–	–	0.4
106	Germacrene-D-4-ol	1574	2058	0.4	0.3
107	$\beta$ -Copaene-4- $\alpha$ -ol	1579	2135	0.4	0.3
<b>108</b>	<b>Spathulenol</b>	<b>1585</b>	<b>2125</b>	<b>8.6</b>	<b>8.6</b>
109	Gleenol	1589	2038	–	2.2
<b>110</b>	<b>Caryophyllene oxide</b>	<b>1591</b>	<b>1991</b>	<b>7.5</b>	<b>10.6</b>
111	Viridiflorol	1596	2080	0.2	0.3
112	Longiborneol	1599	2157	0.4	0.5
113	$\alpha$ -Humulene oxide	1603	2019	0.1	0.1
114	$\beta$ -Atlantol	1607	2012	0.5	0.6
115	Humulene epoxide II	1612	2045	0.4	0.6
116	Tetradecanal	1615	–	0.6	0.7
117	1- <i>epi</i> -Cubebol	1617	–	0.8	0.8
118	Acora-2,4 (15)-dien-11-ol	1625	–	0.2	0.2
119	10- <i>epi</i> -Acora-3,5-dien-11-ol	1629	–	–	0.2
120	$\alpha$ -Acorenol	1635	2161	0.3	0.7
121	<i>allo</i> -Aromadendrene oxide	1638	2008	0.6	0.9
122	<i>epi</i> - $\alpha$ -Muurole	1645	2183	1.9	0.6
123	$\tau$ -Cadinol	1647	–	0.7	2.6
124	$\beta$ -Eudesmol	1652	2223	0.8	1.0
125	11- <i>epi</i> -6,10-Epoxybisabol-3-en-12-al	1656	–	0.2	0.3
126	$\alpha$ -Cadinol	1661	2393	0.9	3.6

**Table 1** (continued)

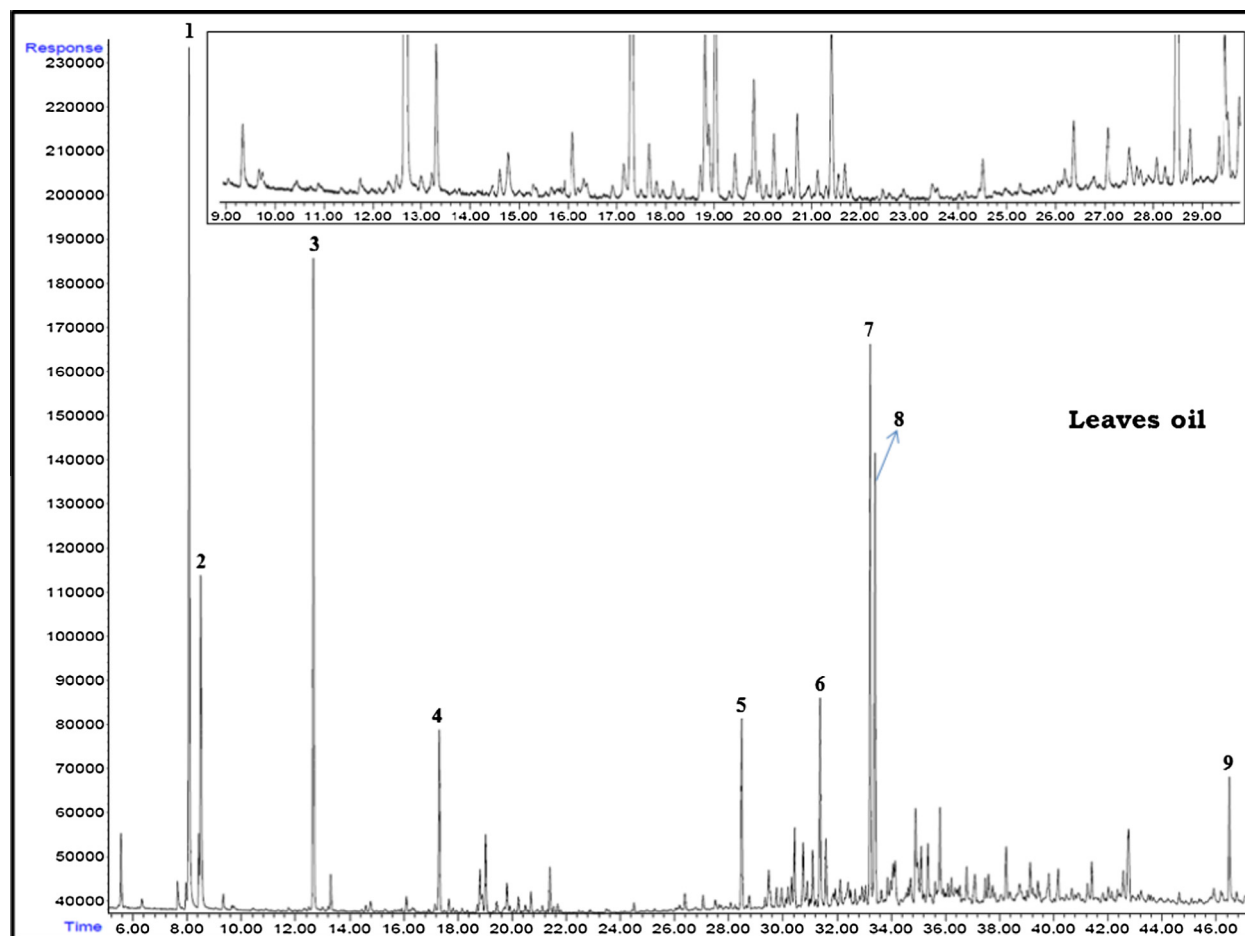
Sl. No.	Compound*	LRI <sup>a</sup>	LRI <sup>P</sup>	LCL (%)	LCF (%)
127	<i>cis</i> -Calamenene-10-ol	1665	2315	0.3	0.4
128	Tridecanoic acid	1671	2613	0.4	0.4
129	<i>trans</i> -Calamenene-10-ol	1674	2341	0.2	0.4
130	$\beta$ -Bisabolol	1677	2142	1.9	0.7
131	Cadalene	1682	2211	0.3	0.4
132	<i>epi-<math>\alpha</math></i> -Bisabolol	1686	–	–	0.3
133	$\alpha$ -Bisabolol	1688	2222	0.4	0.5
134	<i>cis</i> -Apritone	1693	2144	0.6	0.5
135	( <i>Z,Z</i> )-Farnesol	1695	2322	–	0.4
136	<i>n</i> -Heptadecane	1702	1700	0.3	–
137	10- <i>nor</i> -Calamenene-10-one	1705	2353	0.3	0.4
138	<i>trans</i> -Apritone	1714	–	0.6	0.4
139	<i>cis</i> -Nuciferal	1718	–	0.2	0.2
140	( <i>Z,E</i> )-Farnesol	1726	2366	0.7	0.6
141	<i>trans</i> -Nuciferal	1730	–	0.1	0.4
142	Oplopanone	1745	2474	0.5	0.7
143	Xanthorrhizol	1750	–	0.4	0.4
144	<i>trans</i> -Nuciferol	1755	–	–	0.5
145	Tetradecanoic acid	1770	2689	1.3	2.9
146	8,8-Dimethyl-9-methylene-1,5-cycloundecadiene	1775	–	0.4	0.5
147	14-Hydroxy- $\alpha$ -muurolene	1783	2103	–	0.2
148	<i>n</i> -Octadecane	1800	1800	0.3	0.4
149	Hexadecanal	1818	2131	0.2	0.4
150	Avocadynofuran	1825	1938	–	0.6
151	<i>n</i> -Nuciferyl acetate	1832	–	1.0	0.9
152	Eudesm-7(11)-en-4-ol, acetate	1848	–	–	0.1
153	( <i>Z,Z</i> )-Farnesyl acetone	1850	–	–	0.4
154	Pentadecanoic acid	1871	–	0.2	0.2
155	Nonadecane	1900	1900	0.1	0.4
156	Heptadecane-2-one	1902	2232	0.1	0.4
157	( <i>E,E</i> )-Farnesyl acetone	1920	2378	0.4	0.3
158	<i>cis</i> -Hexadec-9-enoic acid	1952	–	1.5	0.1
159	Palmitic acid	1958	–	0.3	0.6
160	<i>n</i> -Eicosane	1999	2000	0.1	–
161	Phytol	2119	2620	2.6	0.3
162	Linoleic acid	2143	–	0.2	0.3
163	Methyloctadecanoate	2147	2429	0.2	–
<i>Class composition</i>					
Monoterpene hydrocarbons				0.5	0.2
Oxygenated monoterpenes				9.0	1.7
Sesquiterpene hydrocarbons				13.5	34.2
Oxygenated sesquiterpenes				35.4	51.0
Aliphatic hydrocarbons				1.6	1.8
Oxygenated aliphatic hydrocarbons				33.5	6.1
Others				2.8	0.3
Total identified				96.3	95.3
Oil yield (%; w/w-fresh weight basis)				0.06	0.08

\* Components are listed in their order of elution from HP-5 MS column; LRI<sup>a</sup> = determined linear retention index on HP-5 MS column; LRI<sup>P</sup> = determined linear retention index on DB-wax column; LCL = *L. camara* leaves oil; LCF = *L. camara* flowers oil; compounds higher than 5.0% are highlighted with boldface; t = trace (<0.05%).

FID using both polar (DB-Wax) and nonpolar (HP-5MS) columns under the same conditions as described above. The detector temperature was maintained at 300 °C for both polar and nonpolar analyses. The relative composition of the oil components was calculated on the basis of the GC–FID peak areas measured using the HP-5 MS column without using correction factor. Results are reported in Table 1 according to their elution order on the HP-5MS column.

### 2.5. Retention indices

A mixture of a continuous series of straight-chain hydrocarbons, C8–C31 (C8–C20, 04070, Sigma–Aldrich, USA and C20–C31, S23747, AccuStandard, USA) was injected into both polar (DB-Wax) and nonpolar (HP-5MS) columns under the same conditions previously described for the oil samples to obtain the linear retention indices (LRIs) (also referred to as



**Figure 1** GC–FID chromatogram of leaves essential oil of *Lantana camara* on HP-5MS column (peaks: 1: *cis*-3-hexen-1-ol; 2: 1-hexanol; 3: 1-octen-3-ol; 4: linalool; 5:  $\beta$ -caryophyllene; 6:  $\gamma$ -cadinene; 7: spathulenol; 8: caryophyllene oxide; 9: phytol).

linear temperature programmed retention indices [LTPRI]) of the oil constituents provided in Table 1. The LRIs were computed using van den Dool and Kratz's equation.

### 2.6. Identification of volatile components

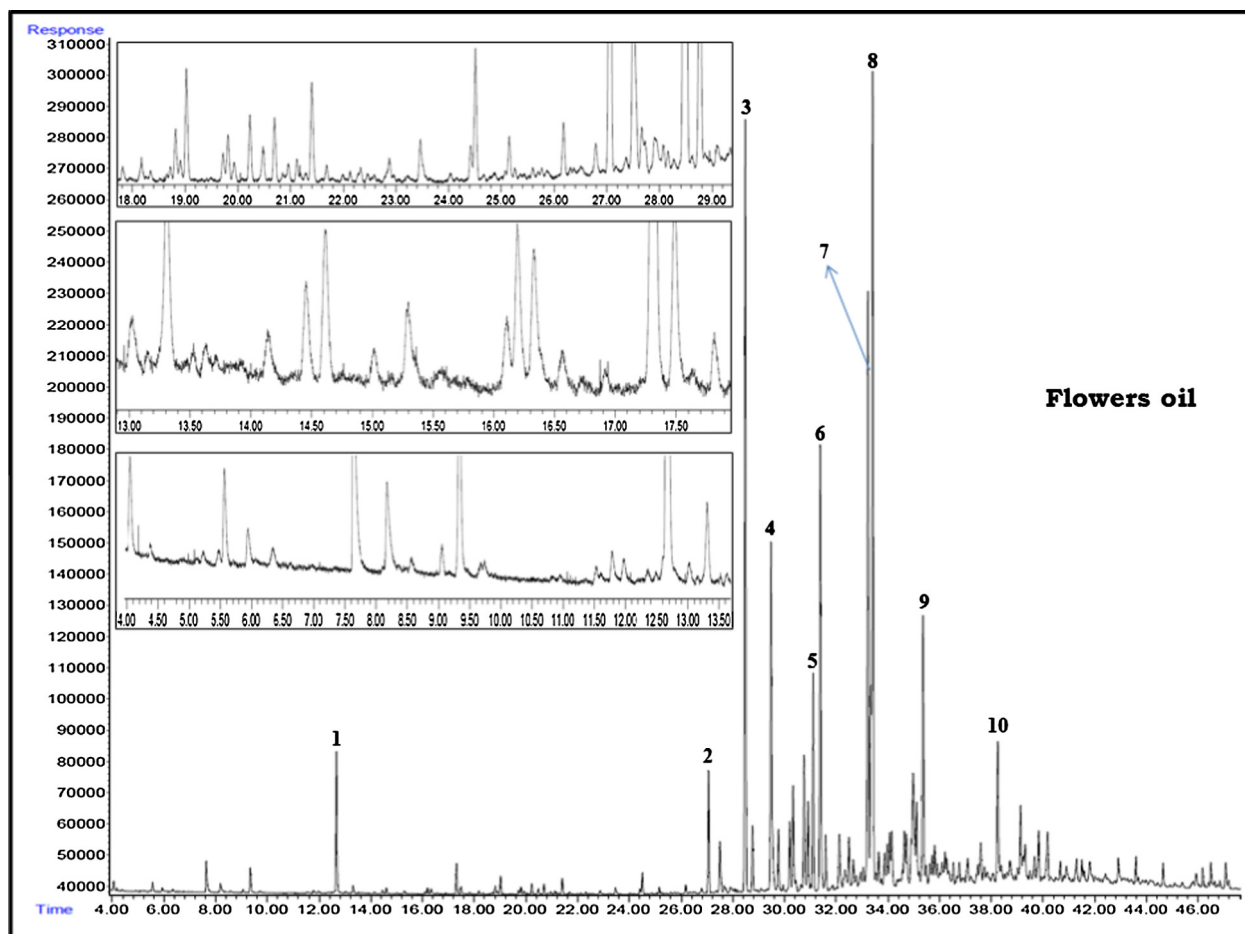
GC–FID chromatogram of leaves and flowers essential oils of *L. camara* with identified peaks of major components on HP-5MS column is shown in Figs. 1 and 2, respectively. The identification of components was done by matching their mass spectra with the library entries (WILEY 9th edition, NIST-08 MS library version 2.0 f as well as the Adams and Flavor libraries) of a mass spectra database as well as by comparing their mass spectra and linear retention indices (LRI) with published data obtained using both polar and nonpolar columns (Acree and Arn, 2015; Adams, 2007; Babushok et al., 2011; Davis, 1990; El-Sayed, 2015; NIST 2015) and the co-injection of authentic standards available in our laboratory.

## 3. Results and discussion

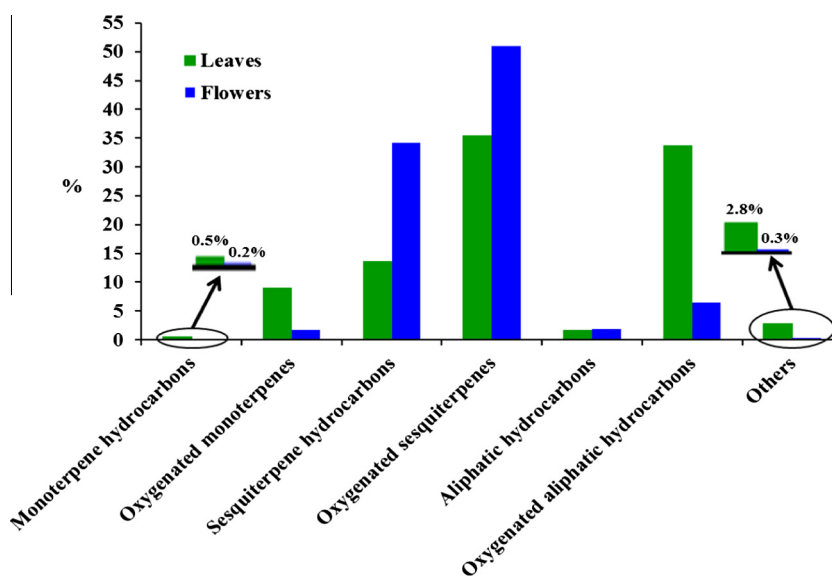
This study describes for the first time detailed characterization of the essential oil constituents derived from leaves and flowers of *L. camara* growing in Saudi Arabia. The hydro-distillation

of *L. camara* leaves and flowers in a Clevenger-type apparatus afforded light-orange color oils in the yield of 0.06% and 0.08%, w/w, respectively, on the fresh weight basis. The phytochemical analysis of leaves and flowers essential oils of *L. camara* was performed on gas chromatography–mass spectrometry (GC–MS) and gas chromatography–flame ionization detector (GC–FID) using both polar and nonpolar columns which resulted in the identification of a total of 163 compounds from leaves and flowers oils, in which 98 compounds were found common in both oils and 36 components were specific to leaves oil whereas 29 components were detected only in flowers oil. In the leaves oil of *L. camara*, 134 compounds were identified, while 127 compounds were identified in the oil obtained from flowers accounting for 96.3% and 95.3% of the total oil compositions, respectively. The identified compounds and their relative contents are listed in Table 1 according to their elution order on a nonpolar HP-5MS column.

Table 1 reveals that the oil from leaves of *L. camara* was dominated by oxygenated sesquiterpenes (35.4%) followed by oxygenated aliphatic hydrocarbons (33.5%), sesquiterpene hydrocarbons (13.5%) and oxygenated monoterpenes (9.0%). Other classes of compounds such as monoterpene hydrocarbons, aliphatic hydrocarbons and others were not present in appreciable amount and account for only 4.9%. On the other hand, the oil from flowers was dominated by



**Figure 2** GC-FID chromatogram of flowers essential oil of *Lantana camara* on HP-5MS column (peaks: 1: 1-octen-3-ol; 2:  $\alpha$ -copaene; 3:  $\beta$ -caryophyllene; 4: *trans*- $\beta$ -farnesene; 5:  $\beta$ -bisabolene; 6:  $\gamma$ -cadinene; 7: spathulenol; 8: caryophyllene oxide; 9:  $\alpha$ -cadinol; 10: tetradecanoic acid).



**Figure 3** Compound classes found in the oils obtained from leaves and flowers of *Lantana camara*.

**Table 2** Major components of *Lantana camara* essential oils reported from various regions of the world.

Geographic regions	Major compounds (%)	References
<b>Cameroon</b>	<i>ar</i> -Curcumene (24.7 <sup>a</sup> ), <b><math>\beta</math>-caryophyllene</b> (13.3 <sup>d</sup> ), caryophyllene epoxide II (7.1 <sup>d</sup> )	Ngassoum et al. (1999)
<b>Egypt</b>	<b><math>\beta</math>-Caryophyllene</b> (15.6 <sup>a</sup> ), $\alpha$ -humulene (9.2 <sup>a</sup> ), bicyclogermacrene (6.7 <sup>a</sup> ), germacrene-D (5.2 <sup>a</sup> ), Farnesol (6.4 <sup>a</sup> ), spathulenol (6.0 <sup>a</sup> )	Elansary et al. (2012)
<b>Nigeria</b>	Sabinene (19.6 <sup>a</sup> , 21.5 <sup>b</sup> ), 1,8-cineole (14.8 <sup>a</sup> , 12.6 <sup>b</sup> ), <b><math>\beta</math>-caryophyllene</b> (12.7 <sup>a</sup> , 13.4 <sup>b</sup> ), $\alpha$ -humulene (6.3 <sup>a</sup> , 5.8 <sup>b</sup> )	Kasali et al. (2004)
<b>South China</b>	Germacrene-D (15.9 <sup>c</sup> ), <b><math>\beta</math>-caryophyllene</b> (12.4 <sup>c</sup> ), $\alpha$ -humulene (9.3 <sup>c</sup> ), germacrene-B (6.2 <sup>c</sup> )	Sundufu and Shoushan (2004)
<b>Iran</b>	<b><math>\beta</math>-Caryophyllene</b> (25.3 <sup>d</sup> ), sabinene (20.2 <sup>d</sup> ), bicyclogermacrene (13.3 <sup>d</sup> ), $\alpha$ -humulene (8.4 <sup>d</sup> ), 1,8-cineole (8.0 <sup>d</sup> )	Sefidkon (2002)
<b>Algeria</b>	<b><math>\beta</math>-Caryophyllene</b> (35.7 <sup>a</sup> ), caryophyllene oxide (10.0 <sup>a</sup> ), $\beta$ -elemene (6.4 <sup>a</sup> )	Zoubiri and Baalioamer (2012a)
<b>Cuba</b>	<i>E</i> -nerolidol (43.4 <sup>a</sup> ), $\delta$ -cadinene (7.6 <sup>a</sup> ), $\alpha$ -humulene (4.9), <b><math>\beta</math>-caryophyllene</b> (4.8 <sup>a</sup> )	Pino et al. (2004)
<b>Congo</b>	<b><math>\beta</math>-Caryophyllene</b> (20.6 <sup>a</sup> ), $\alpha$ -humulene (10.6 <sup>a</sup> ), bicyclogermacrene (8.6 <sup>a</sup> )	Ouamba et al. (2006)
<b>Madagaskar</b>	<b><math>\beta</math>-Caryophyllene</b> (11.3–13.6 <sup>e</sup> , 25.8–30.8 <sup>f</sup> , 15.9 <sup>g</sup> ), davanone (22.6–25.9 <sup>e</sup> , 0.6 <sup>f</sup> , 12.4 <sup>g</sup> ), sabinene (9.4–11.3 <sup>e</sup> , 9.0–14.3 <sup>f</sup> , 14.1 <sup>g</sup> ), linalool (4.8–6.1 <sup>e</sup> , 0.4–1.4 <sup>f</sup> , 5.4 <sup>g</sup> ), $\alpha$ -humulene (4.4–5.2 <sup>e</sup> , 2.4–2.6 <sup>f</sup> , 0.0 <sup>g</sup> )	Randrianalijaona et al. (2005)
Ngaoundere	Davanone (15.9 <sup>d</sup> ), <b><math>\beta</math>-caryophyllene</b> (12.0 <sup>d</sup> ), sabinene (9.0 <sup>d</sup> )	Ngassoum et al. (1999)
Antananarivo	<b><math>\beta</math>-Caryophyllene</b> (18.8 <sup>d</sup> ), $\delta^3$ -carene (9.0 <sup>d</sup> )	Mollenbeck et al. (1997)
<b>Brazil</b>	<b><math>\beta</math>-Caryophyllene</b> (16.2 <sup>a</sup> ), germacrene-D (28.6 <sup>a</sup> ), bicyclogermacrene (14.7 <sup>a</sup> ), germacrene-D-4-ol (19.9 <sup>a</sup> )	de Oliveira et al. (2008)
Crato	Bicyclogermacrene (26.1 <sup>a</sup> ), <b><math>\beta</math>-caryophyllene</b> (19.7 <sup>a</sup> ), germacrene-D (19.2 <sup>a</sup> ), valencene (12.0 <sup>a</sup> ), $\gamma$ -elemene (5.4 <sup>a</sup> )	Sousa et al. (2012)
Vicosa	<b><math>\beta</math>-Caryophyllene</b> (24.4 <sup>a</sup> ), germacrene-D (19.8 <sup>a</sup> ), bicyclogermacrene (11.7 <sup>a</sup> ), $\alpha$ -humulene (9.3 <sup>a</sup> )	Passos et al. (2012)
<b>India</b>		
Lucknow	Germacrene-D (20.5 <sup>a</sup> , 10.6 <sup>b</sup> ), $\beta$ -elemene (7.3 <sup>a</sup> , 14.5 <sup>b</sup> ), $\gamma$ -elemene (10.3 <sup>a</sup> , 6.8 <sup>b</sup> ), <b><math>\beta</math>-caryophyllene</b> (9.4 <sup>a</sup> , 7.0 <sup>b</sup> ), $\alpha$ -copaene (5.0 <sup>a</sup> , 10.0 <sup>b</sup> ), $\alpha$ -cadinene (3.3 <sup>a</sup> , 7.2 <sup>b</sup> )	Khan et al. (2002)
Dibrugarh	Davanone (47.8 <sup>a</sup> , 7.4 <sup>b</sup> ), <b><math>\beta</math>-caryophyllene</b> (10.3 <sup>a</sup> , 26.9 <sup>b</sup> ), bicyclogermacrene (4.9 <sup>a</sup> , 12.5 <sup>b</sup> ), $\delta$ -cadinene (2.9 <sup>a</sup> , 7.4 <sup>b</sup> )	Misra and Saikia (2011)
Kumaun	Germacrene-D (27.9 <sup>c</sup> ), germacrene-B (16.3 <sup>c</sup> ), <b><math>\beta</math>-caryophyllene</b> (9.6 <sup>c</sup> ), $\alpha$ -humulene (5.8 <sup>c</sup> )	Padalia et al. (2010)
Dehradun	<b><math>\beta</math>-Caryophyllene</b> (23.3 <sup>a</sup> ), $\alpha$ -humulene (11.5 <sup>a</sup> ), germacrene-D (10.9 <sup>a</sup> ), davanone (7.3 <sup>a</sup> )	Rana et al. (2005)

<sup>a</sup> Leaves oil.<sup>b</sup> Flowers oil.<sup>c</sup> Aerial parts oil.<sup>d</sup> Leaves and flowers oil.<sup>e</sup> Oil of aerial parts with pink-violet flowers.<sup>f</sup> Oil of aerial parts with yellow-orange flowers.<sup>g</sup> Industrial oil.

oxygenated sesquiterpenes (51.0%) followed by sesquiterpene hydrocarbons (34.2%) and oxygenated aliphatic hydrocarbons (6.1%). Other chemical classes including monoterpene hydrocarbons, aliphatic hydrocarbons, and oxygenated monoterpenes contributed to only 4.0% (Fig. 3).

The major constituents of leaves oil were *cis*-3-hexen-1-ol (11.3%), 1-octen-3-ol (8.7%), spathulenol (8.6%), caryophyllene oxide (7.5%) and 1-hexanol (5.8%), while the main compounds of the oil from flowers were caryophyllene oxide (10.6%),  $\beta$ -caryophyllene (9.7%), spathulenol (8.6%),  $\gamma$ -cadinene (5.6%) and *trans*- $\beta$ -farnesene (5.0%).

A comparison between leaves and flowers oils of *L. camara* based on chemical classes reveals that the oxygenated sesquiterpenes and oxygenated aliphatic hydrocarbons were the most prevalent groups in leaves oil, accounting for 68.9% of the total oil compositions, whereas, in the flowers oil oxygenated sesquiterpenes and sesquiterpene hydrocarbons were the most dominating chemical groups, accounting for 85.2% of the total

oil compositions. This advocates that both oils contain oxygenated sesquiterpenes as most dominating class of compounds. Nevertheless, the two oils could be easily differentiated from each other considering the amounts of sesquiterpene hydrocarbons and oxygenated aliphatic hydrocarbons. In the flowers oil, content of sesquiterpene hydrocarbons was 2–3 times more than that in the leaves oil, whereas, the content of oxygenated aliphatic hydrocarbons was found to be 5–6 times more in leaves oil than that in flowers oil.

Furthermore, the data presented in Table 1 also suggest that leaves and flowers oil of *L. camara* showed some important qualitative similarities, since out of 163 components identified from both oils, 98 compounds (73.7% in leaves oil and 87.2% in flowers oil) were found to be common in both oils, although they differed significantly with one another in terms of their relative concentrations. For example, the amount of linalool and phytol was 9–10 folds more in leaves oil than that in the oil from flowers, whereas 1-octen-3-ol, *epi*- $\alpha$ -muurolol



and  $\beta$ -bisabolol were found to be 2–5 folds more in leaves oil. Conversely, the amount of *trans*- $\beta$ -farnesene,  $\alpha$ -curcumene and  $\alpha$ -cadinol was 4–7 times greater in the oil from flowers than in the oil from leaves, while the amount of  $\beta$ -caryophyllene,  $\beta$ -bisabolene,  $\tau$ -cadinol, tetradecanoic acid and *epi*-cubebol was 2–3 folds more in flowers oil. Moreover, it is significant to note that two oxygenated aliphatic hydrocarbons, *cis*-3-hexen-1-ol (11.3%) and 1-hexanol (5.8%) identified in leaves oil as major components were not present in flowers oil of *L. camara*. Importantly, to the best of our knowledge, these two components, *cis*-3-hexen-1-ol and 1-hexanol are identified for the first time in *Lantana* oil. *cis*-3-Hexen-1-ol, famously known as leaves alcohol widely used in flavors and fragrances industries for imparting fresh green leafy aroma to various products (Vasiliev et al., 2003). It is found in essential oils of many plants but often in low concentration and thus many synthetic procedures have been attempted for the synthesis of this commercially important compound (Moreno-Marrodan et al., 2012). Moreover, *cis*-3-hexen-1-ol and 1-hexanol have been reported to possess potent inhibitive properties against fusarium diseases (Cruz et al., 2012).

It is noteworthy to mention here that other secondary metabolites particularly, spathulenol,  $\beta$ -caryophyllene and caryophyllene oxide that were identified as major components in the essential oils of present study have been demonstrated to have various important biological activities and industrial applications. For example, spathulenol, an oxygenated sesquiterpene is known for its immunomodulatory and MDR reversal activities (Martins et al., 2010; Ziaei et al., 2011). It is also used as an important ingredient in perfumery, food, pharmaceutical, detergent and cosmetic industries (Leendert et al., 1988), whereas,  $\beta$ -caryophyllene, a bicyclic sesquiterpene with a rare cyclobutane ring and its epoxide derivative caryophyllene oxide have shown numerous important biological activities including neuroprotective, anesthetic, antitumor, immunomodulatory, anti-inflammatory, anticancer, antiviral, anti-mutagenic, anti-proliferative and analgesic activities (Assis et al., 2014; Astani et al., 2011; Chang et al., 2013; Sabulal et al., 2006; Sarpietro et al., 2015). Furthermore, since both compounds possess woody and spicy aroma they are frequently used as flavors and fragrances in various food products and beverages, in soap, lotions, creams, and also in spice blends and citrus flavors and are included in the European list of flavoring substances (Anonymous, 2012; Sabulal et al., 2006; Sarpietro et al., 2015).

Comparison of chemical compositions of leaves and flowers essential oils of *L. camara* growing in Saudi Arabia with those previously studied from different parts of the world (Filho et al., 2012; Kasali et al., 2004; Khan et al., 2002; Love et al., 2009; Ngassoum et al., 1999; Padalia et al., 2010; Sefidkon, 2002; Sundufu and Shoushan, 2004) revealed that the oil compositions determined in the present study differed significantly from those reported earlier (Table 2). For example, *cis*-3-hexen-1-ol and 1-hexanol that were determined as major components in the present study have not been detected earlier in any *L. camara* essential oils analyzed up to now. In contrast, germacrene-D, a natural sesquiterpene hydrocarbon, which has been identified as one of the major components in most of the *L. camara* oils, was detected in trace amount in the present study.

It is significant to mention here that the chemical composition of *L. camara* essential oils studied until now from different

regions of the world has shown prodigious variations (see Table 2). However, it has been noticed that  $\beta$ -caryophyllene, a natural bicyclic sesquiterpene was the only compound that was found either as a major or in appreciable amount in all the *L. camara* essential oils studied so far. Thus,  $\beta$ -caryophyllene could be used as a chemical marker for the *Lantana* essential oils.

#### 4. Conclusion

*L. camara* essential oils have shown remarkable variations in their chemical compositions in relation to their place of collection. In the present study, essential oils of *L. camara* growing in Saudi Arabia have also shown a distinct composition where *cis*-3-hexen-1-ol and 1-hexanol are major components. To the best of our knowledge, these two components are being reported here for the first time in *Lantana* oils. Moreover,  $\beta$ -caryophyllene, a natural bicyclic sesquiterpene with a rare cyclobutane ring which has been found in all oils of *L. camara* studied so far, was also detected as one of the major components in the present study as well, indicating that  $\beta$ -caryophyllene could be used as a chemical marker for the *Lantana* essential oils. Furthermore,  $\beta$ -caryophyllene and *cis*-3-hexen-1-ol have wide industrial applications, for example,  $\beta$ -caryophyllene is used in soap, lotions, creams, and also in various food products and beverages, and in spice blends and citrus flavors. On the other hand, *cis*-3-hexen-1-ol has a great demand in flavors and fragrances industries for imparting fresh green leafy aroma to various products, considering the fact that  $\beta$ -caryophyllene and *cis*-3-hexen-1-ol are the major components of essential oil of *L. camara* which is abundantly available in Saudi Arabia and hence, can be used as a cheap and renewable source for industrial isolation of  $\beta$ -caryophyllene and *cis*-3-hexen-1-ol.

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