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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; β-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 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%), β -caryophyllene (9.7%), spathulenol (8.6%), γ -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.

1. Introduction

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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

Lantana is a genus of both herbaceous plants and shrubs containing

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, linaroside, 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, nematicidal, 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. KSUHZK-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, α bisabolol, and α -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 splitsplitless injector, a quickswap assembly, an Agilent model 7693 autosampler and a HP-5MS fused silica capillary column (5% phenyl 95% dimethylpolysiloxane, $30 \text{ m} \times 0.25 \text{ mm}$ i.d., film thickness 0.25 µm, Agilent Technologies, USA). Supplementary analyses were performed on a DB-Wax fused silica capillary column (polyethylene glycol, $30 \text{ m} \times 0.25 \text{ mm i.d.}$, film thickness 0.25 µm, 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 μ 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

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