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Chemical characterization of volatile organic compounds (VOCs) of [*Lantana camara* var. *aculeata* (L.) Moldenke] with polar solvent extractions

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ABSTRACT

The present investigation was carried out to identify organic volatiles components present in *Lantana* leaves and found to exert biopesticidal-repellent action on many pests of horticultural crops. Volatile organic chemicals (VOCs) eluted from [*Lantana camara* var. *aculeata* (L.) Moldenke] was extracted ascrude oil and analysed by gas chromatography-mass spectrometry (GC-MS). A total of 10 to 15 volatile compounds present in ethanol and methanol extracts were identified. These compounds were found distributed in several chemical classes namely, alcohols, carbonyl compounds (ketones, aldehydes, and esters), fatty acids, terpenes, nitrogenous and sulphur compounds. Of these various classes of compounds identified,Phenol-2-methyl-5-(1,2,2-trimethylcyclopentyl) - (S)-(CAS)-10.36 per cent and 2- (p-Methylphenyl) benzimidazole-7.86 per cent constituted major groups accounting for ethanol and whereas sulphur compounds *viz.*,stigmast-5-en-3-ol, (3.beta.) 15.48 per cent, caryophyllene (7.63%) and phenol having 14.96 per cent constituted major groups in methanol extracts.

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INTRODUCTION

The verbenaceae family of *Lantana* is native to tropical and subtropical America and has been dispersed throughout the world as a popular ornamental plant and because one of the world's worst weeds [*Lantana camara* var. *aculeata* (L.) Moldenke] (Day *et al.*, 2003) and different parts of this plant are used for medicinal and non-medicinal purposes because of relative amounts of some triterpene ester metabolites. *L. camara* var. *aculeata* is rich in secondary metabolites possessing beneficial biological activities. In India, these plants are used in folk and traditional medicine systemsas antimicrobial, fungicidal, insecticidal and nematicidal agent (Anita and Sawant, 2011). Physico-chemical

properties of pentacyclic-triterpenoids revealed the difficulty in getting pure form of lantadene. Whole extracts of lantadene was completely eluted from the methanol-water mixture. Flower and fruit extracts of lantana contain low percentage of lantadene and qualitative test, spectrophotometric and chromatographic analysis (Geetali and Anita, 2014). The main chemical constituent, lantadeneacts as promoting agentdue to its polymorphism of chemical constituent and its polymorphic forms, differed in melting behaviour (Eralanio *et al*., 2012). The precise mechanism and nature of polymorphic forms have not yet been clear hence, the study was undertaken to determine the chemical profile of the crude extracts/ essential oil of *Lantana* leaves.

MATERIAL AND METHODS

Collection of test plant materials :

Aerial parts of the test plant *L. camara* were collected from Thondamuthur and Aalandurai block of Coimbatore district of Tamil Nadu. The samples were air-dried for 15-20 days under shade. After complete shade drying, the plant parts were pulverized into powder with the help of motor grinder.

Extract preparation- Soxhlet apparatus :

To extract more active principle of plant material was subjected to Soxhlet extraction (Sukthamrong *et al.*, 1981 and Sharma and Gupta, 2009). Known amount (50-75g thimple^{-1}) of plant material of each solvent was filled into the Soxhlet apparatus. A cotton plug was used at the place of thimble to stop the entry of the crude material into the siphoning tube. The required organic solvents *viz.*, ethanoland methanol were filled up five times more than total amount of the sample material into the flask of the apparatus. The apparatus was then connected with the water supply to the condenser. The temperature of the heating mantle was maintained according to the boiling point of respective solvents. The process was carried out for 24 hrs for each sample.

Pooled extracts were filtered using a Whatman filter paper No.1.and concentrated by rotary evaporation at 40°C. After drying in desiccator, crude extracts were weighed, stored in stock vials and kept in refrigerator (4°C) for further use. It was further purified by column chromatography (anhydrous Na_2SO_4 + silica gel + dehydrated charcoal) for GC MS analysis.

Gas chromatography – Mass spectrometry analysis of *L. camara* leaf extract :

GC-MS (Shimadzu QP-2010 plus) analysis was done in pesticide Toxicology laboratory, Department of Agricultural Entomology and following method described by Shettima *et al.* (2013). The GC-MS was equipped with a split injector and an ion-trap mass with 220°C spectrometer detector together with a fused silica capillary column (RXI-1MS) having a thickness of 0.25 µm, dimensions of 30 m x 0.25 mm and temperature limits of 60°C to 260°C. The column temperature was programmed between 60°C and 260°C at a rate of 3.0ml per min. The mass range and temperature of the injector and detector were at 500 M/z of 220°C and 200°C, respectively. Helium gas was used as a carrier gas at a flow rate of 0.95 ml per minute. The chromatogram obtained from the GC was then analysed in the mass spectrography (MS) to get the mass of all the fractions.

Identification of components :

Identification was based on the molecular structure, molecular mass and calculated fragments. Interpretation on mass spectrum GC-MS was conducted using the database of National Institute Standard and Technology (NIST) having more than 62,000 patterns. The name, molecular weight and structure of the components of the test materials were ascertained. Relative percentage amount of each component was calculated by comparing its average peak area to the total areas. The spectrum of the unknown component was compared with the spectrum of the component stored in the NIST library version-2005 Software-Turbomas 5.2. This is done in order to determine whether this plant species contains any individual compound or group of compounds, which may substantiate its current commercial and traditional use as an herbal medicine. Further it helps to determine the most appropriate methods of extracting these compounds. These results are consequently being discussed in the light of their putative biological activities (Sermakkani and Thangapandian, 2012).

RESULTS AND DISCUSSION

The phytochemical components present in the all crude extracts of *Lantana* in different solvents were identified by GC-MS. The results showed the presence of alkaloids, tannins, flavonoids, saponins, steroids and reducing sugar in the plant. Table 1and 2 reveal the groups

of secondary metabolites detected in each solvent fraction. The chromatogram and phyto-chemical components with their retention time, molecular weight and percentage of composition in different solvents extracts are presented in Fig. 1 and 2.

Ethanol leaves extracts :

GC-MS is one of the best techniques to identify the constituents of volatile matter, long chain, branched chain hydrocarbons, alcohols acids, esters etc. The GC-MS analysis of *Lantana* leaves revealed the presence of ten compounds (Fig.1.) that could contribute the biopesticidal-repellent quality of active principles. The identification of the phytochemical compounds was confirmed based on the peak area, retention time and molecular formula. The active principles with their Retention time (RT), Molecular formula, Molecular weight (MW) and peak area in per cent are presented in Table 1. Phenol, 2-methyl-5-(1, 2, 2-trimethyl cyclopentyl) - (S), structure, spectrum (Fig. 6), MWt.-218 Rt-18.08 and10.36 per cent of area; 2-Hexadecen-1-ol, 3,7,11,15-tetramethylstructure, spectrum (Fig.4.) MWt.-296 Rt-

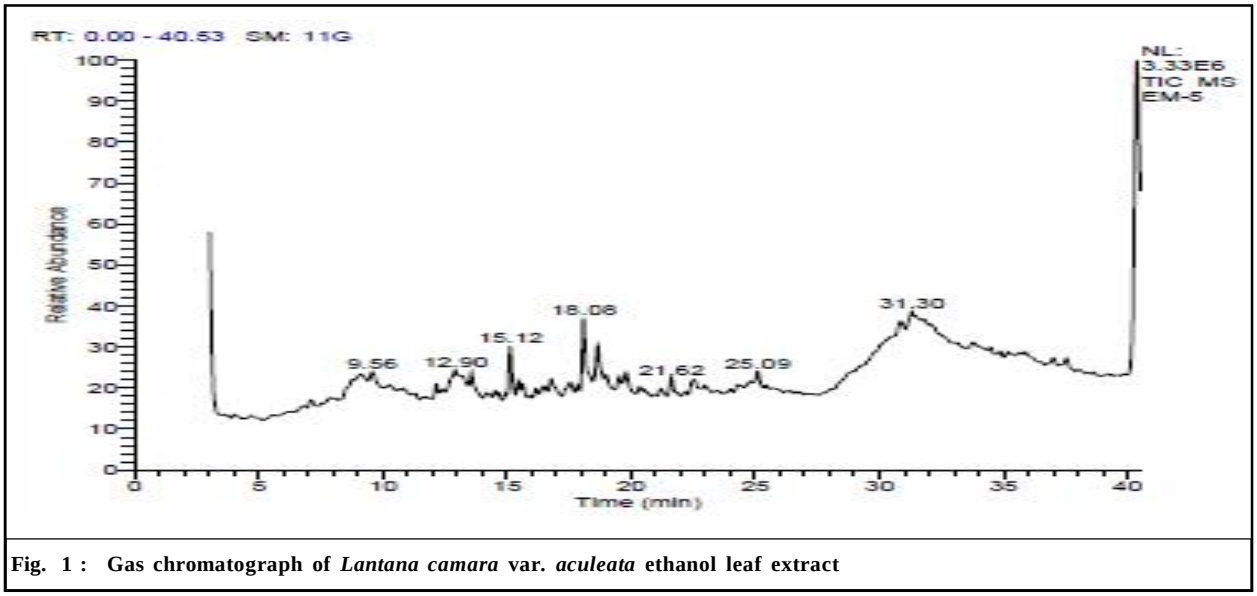

Fig. 1 : Gas chromatograph of *Lantana camara* var. *aculeata* ethanol leaf extract

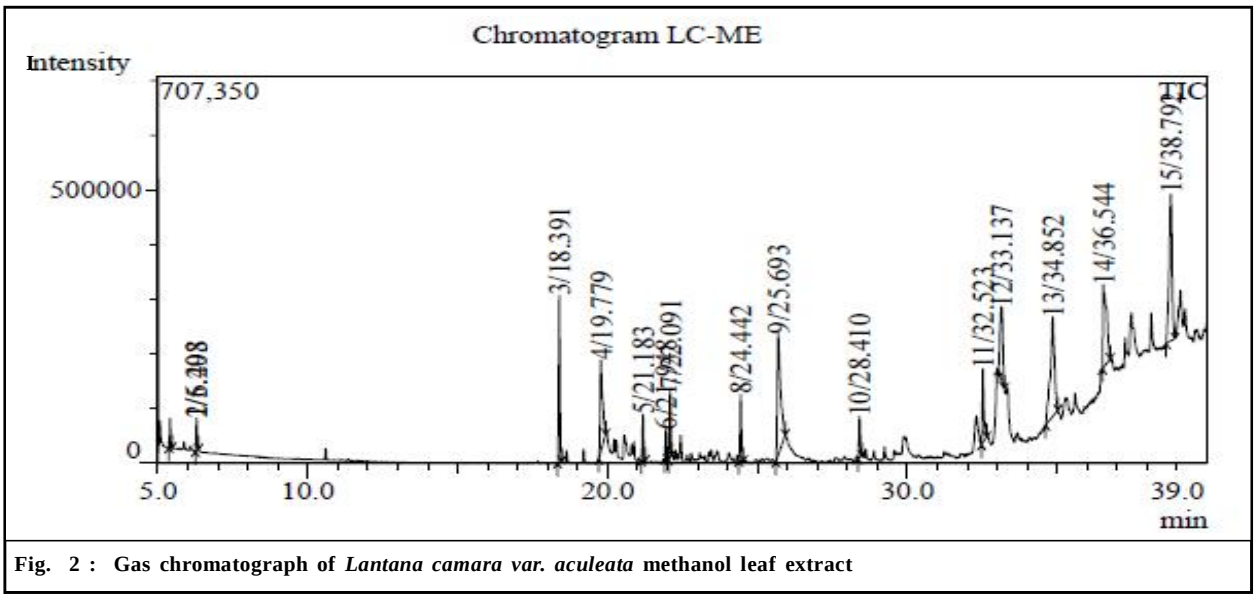

Fig. 2 : Gas chromatograph of *Lantana camara var. aculeata* methanol leaf extract

25.09 and 3.44 per cent of area and 1-(Allenylthio)-2,6-dimethyl-3-vinyl-2-cyclohexene structure and spectrum (Fig.5) were recorded andthey were the major compounds of ethanolcrude extracts. The first compound identified with less retention time (7.10min) was (4à, 4aá, 9aá) -9a - Chloro - 8 - hydroxy - 1-1,4 - trimethoxy -1, 4, 4a, 9a - tetrahydro - anthraquinone, whereas 2-[(2,5 Dichlorophenyl) amino] benzoic acid was the last compound, which took longest retention time (35.91min) to identify.

Methanol leaves extracts :

Methonolic extracts of leaf was subjected to GCMS analysis of the preliminary phyto-chemicals screening showed the presence of phyto-chemicals in the chromatogram (Fig. 2). The probable compounds present in the leaf extracts were searched in the plant compound library with respect to the mass and retention time of each fraction from the column. Cyclopentane-3-spiropentacyclo (16.44%) was identified as the major component followed by Stigmast-5-en-3-ol (15.48%), Phenol,2-methyl-5-(1,2,2-trimethylcyclopentyl)-(S) (Fig.6.) (14.96%), Tetrapentacontane (10.86%) and trans - Caryophyllene (Fig.3.) having (7.63%) and other compounds are of minor quantity and their peak area and molecular weight of the compound have been

Table 1 : Majorphytochemical components identified in the ethanol crude extracts of *Lantana camara var. aculeata* (L.) Moldenke

Peak No.	Retention time	Area %	Molecular weight.	Components
1	7.10	1.06	352	(4à,4aá,9aá)-9a-Chloro-8-hydroxy-1,1,4-trimethoxy-1,4,4a,9a-tetrahydro anthraquinone
2	9.56	1.82	150	4-Methyl-2-(3-methyl-2-butenyl)-furan
3	12.90	7.38	206	1-(Allenylthio)-2,6-dimethyl-3-vinyl-2-cyclohexene
4	15.12	7.86	208	2-(p-Methylphenyl)benzimidazole
5	18.08	10.36	218	Phenol, 2-methyl-5-(1,2,2-trimethyl cyclopentyl)-,(S)- (CAS)
6	21.62	2.85	270	Pentadecanoic acid, 14-methyl-, methyl ester (CAS)
7	25.09	3.44	296	2-Hexadecen-1-ol, 3,7,11,15-tetramethyl-[R-[R*,R*- (E)]](CAS)
8	31.30	6.41	390	1,2-Benzenedicarboxylic acid, bis (2-ethylhexyl) ester (CAS)
9	31.77	4.33	325	3-(1-acetyl-3-indolyl)-2-indolylacrylonitrile
10	35.91	1.80	281	2-[(2,5-Dichlorophenyl) amino] benzoic acid

Table 2 : Major phytochemical components identified in the methanol crude extracts of *Lantana camara var. aculeata* (L.) Moldenke

Peak No.	Retention time	Area %	Molecular weight.	Components
1	5.408	1.13	116	1-Pentanol,
2	6.293	1.38	98	3-Hexen-2-one
3	18.391	7.63	204	trans-Caryophyllene
4	19.779	8.30	208	Benzene
5	21.183	2.01	222	Selina-6-en-4-ol
6	21.948	1.26	220	1H-Cycloprop[e]azulen-7-ol,
7	22.091	3.19	220	(-)-5-Oxatricyclo [8.2.0.0(4,6)] Dodecane
8	24.442	3.21	190	3,7-Cyclodecadien-1-one
9	25.693	14.96	218	Phenol
10	28.410	2.24	208	2,6,10-Trimethyl
11	32.523	4.29	296	2-Hexadecen-1-ol
12	33.137	7.62	204	6S-2,3,8,8-Tetramethyltricyclo [5.2.2.0(1,6)]undec-2-ene
13	34.852	15.48	376	Stigmast-5-en-3-ol, (3.beta.)
14	36.544	10.86	414	Tetrapentacontane
15	38.792	16.44	758	Cyclopentane-3'-spiropentacyclo

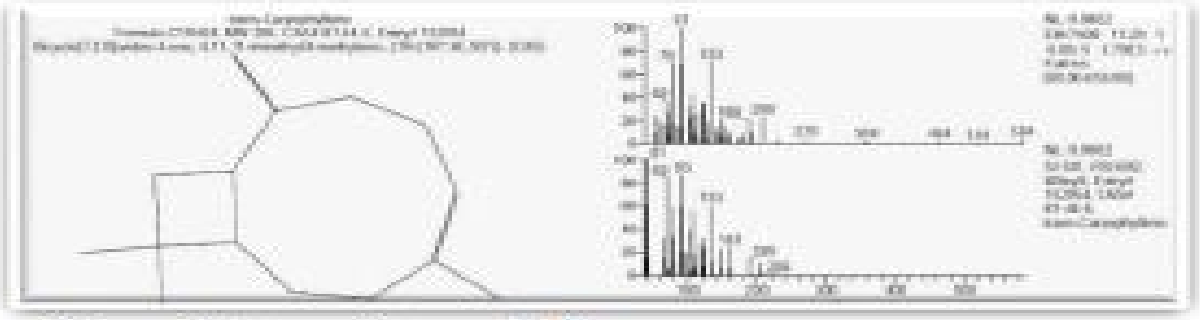

Fig. 3 : Trans-Caryophyllene

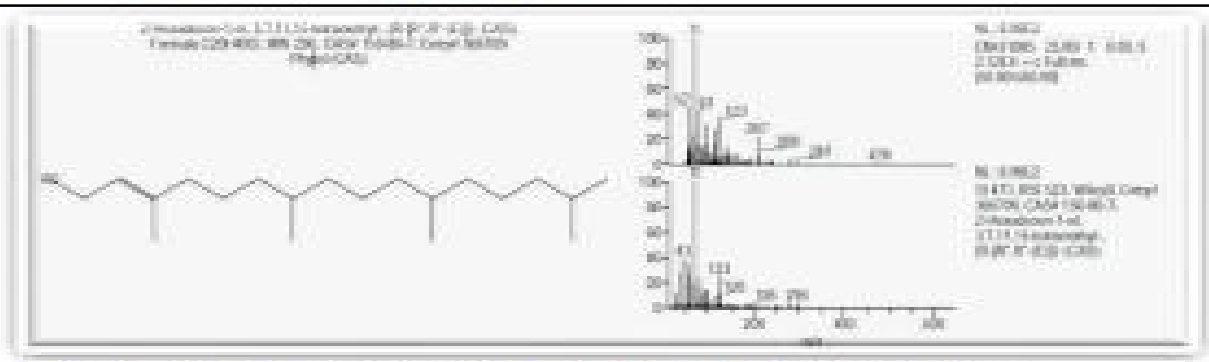

Fig. 4 : 2-Hexadecen-1-ol,3,7,11,15-tetramethyl

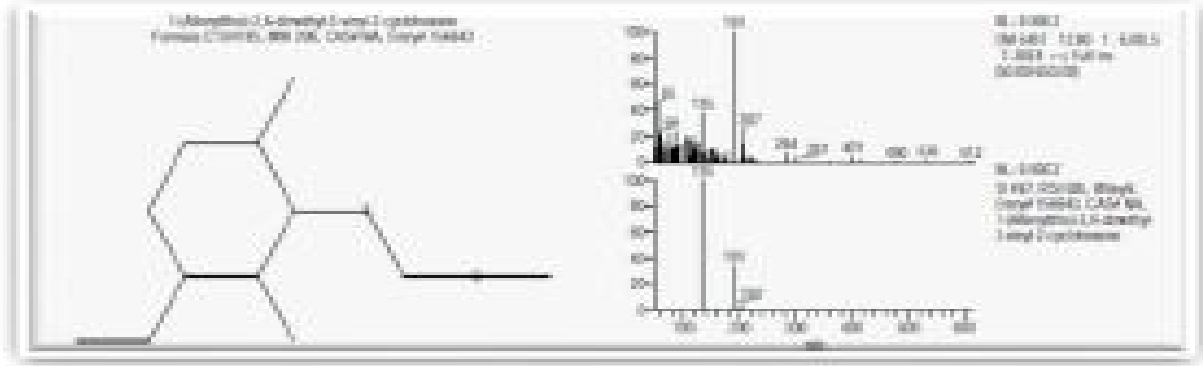

Fig. 5 : 1-(Allenylthio)-2,6-dimethyl-3-vinyl-1cyclohexene

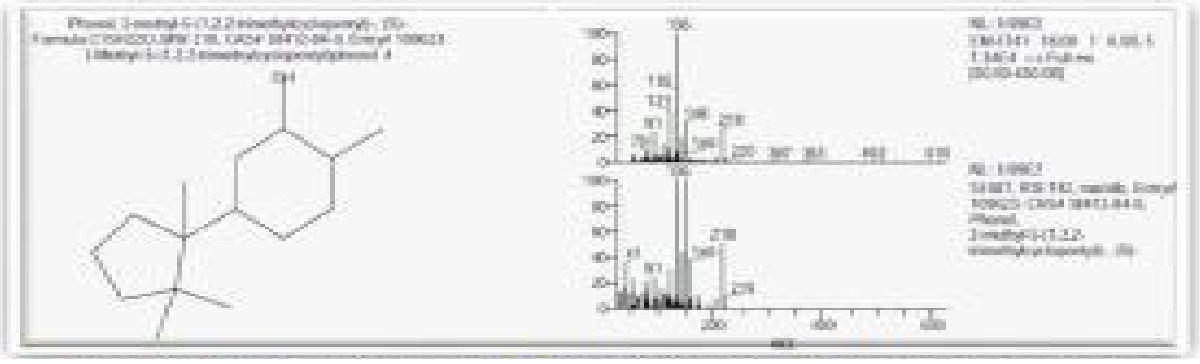

Fig. 6 : Phenol, 2-methyl-5-(1,2,2-trimethylcyclopentyl)-(S)

tabulated in (Table. 2).

Present study indicated caryophyllene contents are present in the methanol extracts, whereas absent in ethanol extracts due to elution of the solvent characteristics and boiling temperature. Earlier attempts to extract the volatile compounds of *Lantana* through soxhlet apparatus followed by concentrate under vacuum drier gave crude extracts in which many volatile constituents were identified (Passos *et al.*, 2012). However, based on the previous reports (Geetali and Anita, 2014) in which it was shown that certain fractions of Lantadene compounds added to *Lantana* leaf could not be detected after extraction of *Lantana* leaves as a crude form in present study. Many volatile components might have either escaped due to their high volatility or had undergone changes in their structure and activity due to the extreme conditions of temperature and pressure employed in the extraction procedure.

The ethanol and methanol extracts of *Lantana* have been subjected to gas chromatography-mass spectroscopy (GC-MS) analysis and 10 and 15 constituents were reported, respectively. Some authors reported more compounds and the essential oil was bright yellow in colour, highly volatile and had eighteen compounds identified representing 100 per cent of the oil (Jawonisi and Adoga, 2013). The constituents of the methanol crude extracts had Caryophyllene and 2-Hexadecen - 1 -ol, 3, 7, 11, 15 - and the same was supported by Jawonisi and Adoga, 2013; Passos *et al.* (2012) and Murugesan *et al*. (2012). Singh *et al*. (1989) indicated that the phytotoxins present in *Lantana* leaf extracts were phenolic in nature and characterization of the phenolic fraction by HPLC revealed the presence of 14 phenolic compounds listed as protocatechuic acid, gentisicacid, p-hydroxybenzoic acid, vanillicacid, caffeic acid, syringic acid, vanillin, p-coumaric acid, m-coumaric acid, ferulic acid, salicylic acid, o-coumaric acid, t-cinnamic acid andmethyl coumarin (Jain *et al*., 1989).

Conclusion:

In the present study different chemical constituents of *Lantana* leaves were identified byethanol and methanol extracts through Gas chromatogram mass spectrometry (GC-MS) analysis. *Lantana* had significantly different chemical composition and the oil produced by *Lantana* contained a greater diversity of compounds. In the present study crude extracts are known to possess anti feedant, repellency, insect growth regulation and strong oviposition deterrencey. Further investigations have to be carryout to find the activity of crude extracts and to incorporate the IPM schedule. This trend is in line with the requirements of new regulations on integrated pest management in vegetable ecosystems.

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