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Original scientific paper

Chemical constituents of the essential oils of *Tephrosia purpurea* and *Ipomoea carnea* and their repellent activity against *Odoiporus longicollis*

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Abstract: The chemical constituents of the essential oils (EOs) obtained from stem and root of *Tephrosia purpurea* (L.) Pers. and *Ipomoea carnea* Jacq. were investigated by gas chromatography–mass spectrometry (GC–MS). The total contents of lipid and oil were higher in the stem than in the root of *T. purpurea* and *I. carnea.* The essential oils extracted from the stem and root of *T. purpurea* and *I. carnea* contained 9 and 8 compounds, respectively. Hexadecanoic acid was found to be the principal constituent of stem (69.61 %) and root (46.97 %) of *T. purpurea* and of the stem (70.61 %) and root (88.89 %) of *I. carnea*. The findings of the present study suggest that the EOs of *T. purpurea* and *I. carnea* could be a source of hexadecanoic acid that could be used for industrial purposes. The essential oils of *T. purpurea* and *I. carnea* showed stronger repellent activity for males (–0.73 and –0.70 for *T. purpurea* and *I. carnea* stem EO, respectively) than for females (–0.63 and –0.59 for *T. purpurea* and *I. carnea* stem EO, respectively) of the banana pseudostem weevil *Odoiporus longicollis.* The results indicated that the active compounds of essential oils from stems of *T. purpurea* and *I. carnea* could be explored as natural repellents for the control of *Odoiporus longicollis.*
Keywords: *Tephrosia purpurea*; *Ipomoea carnea*; essential oils; hexadecanoic acid; repellent activity.

INTRODUCTION

Essential oils are complex mixtures of volatile compounds characterized by a strong odor, and are utilized in pest control measures. *Tephrosia purpurea* (Linn.) Pers (Fabaceae) is a copiously branched, herbaceous, perennial and medi-

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cinally important plant.[1] It possesses antitumor[2] antiplasmodial[3] and anti-diabetic[4,5] activities. *T. purpurea* extracts showed insecticidal activity against houseflies, mosquitoes, rice weevil and flour beetle[6] *Myzus persicae* adults, third instar larvae of *Pluttella xylostella*[7] and adults of *Dysdercus cingulatus* (Fab.).[8] Phytochemical investigations of *T. purpurea* indicated the presence of terpurinflavone,[3] semiglabrin, pongamol, lanceolatins A and B, rutin, lupeol, and *β*-sitosterol. Flavonoids including (+)-tephrorin A and B, (+)-tephrosone; 7,4′--dihydroxy-3′,5′-dimethoxyisoflavone (isoflavone) and a chalcone, (+)-tephropurpurin,[9] tephroglabrin, tepurindiol,[10] apollinine, tephropurpulin A[11] and isoglabratephrin[12] were reported to be present.

Ipomoea carnea Jacq. (Convolvulaceae) is a common weed as well as a toxic plant found abundantly in India, Brazil, USA, *etc*.[13] Different extracts of *I. carnea* possess antibacterial, antifungal, anti-oxidant, anticancer, anticonvulsant, immunomodulatory, hepatoprotective, anti-inflammatory, anxiolytic, sedative, wound healing[14,15] and skin disease healing activities.[16] This weed also has depressant,[17] HIV type 1 reverse transcriptase inhibitory[18] and antidiabetic activities.[19] The toxic principles of this plant include calystegines B2, C1, swainsonine[20] 2-epi-lentiginosine, calystegines B1, B2, B3, C1, and *N*-methyl-*trans*-4--hydroxy-L-proline,[21] whereas non-toxic principles, such as chitinase, were recorded.[13,22] Various solvent extracts of *Ipomoea carnea* had ovicidal activity against the gelechiid *Phthorimaea operculella*[23] and insecticidal activity on *Achaea janata.*[24]

Odoiporus longicollis (Oliv.) (Insecta: Coleoptera: Curculionidae) or banana stem weevil (BSW) is one of the most widely spread and serious monophagous pests of banana. It has been estimated that the BSW causes 10–90 % yield losses depending on the infestation stage and management efficiency. Control of the BSW relies heavily on the use of synthetic insecticides, which has led to several adverse effects on the environment, induced insect resistance and toxicity to non-target species.[25] Hence, botanical insecticides, such as neem azal,[25] azadirachtin[26] and Gin–Dulaw–Luya[27] (a combination of ginger, turmeric and gin) were tested for their efficacy against this weevil to develop an alternative control method that is safe for the environment and highly efficient for the management of *O. longicollis.*

The essential oils of these plants, with ovicidal, larvicidal, repellent, deterrent, antifeedant and toxic effects against a wide range of insects have the potential for use in crop protection.[28–31] There are no reports on the chemical constituents and repellent properties of *T. purpurea* and *I. carnea* essential oils. This paper presents the first report on the essential oils of these plants and their chemical composition as well as repellent activity against *O. longicollis* adults.

MATERIALS AND METHODS

Plant materials

Root and stem of *T. purpurea* and *I. carnea* were collected from in and around Palayamkottai (8.7166° N and 77.7333° E). The plant materials were identified by the Department of Plant Biology and Plant Biotechnology, St. Xavier's College (Autonomous), Palayamkottai. Voucher specimens were deposited with the Crop Protection Research Centre, St. Xavier's College, Palayamkottai (herbarium specimen number: CPRC TP-006 and CPRC TP-007 for *T. purpurea* and *I. carnea* respectively). Immediately after collection, the stem and root portions were separated and air-dried for two weeks under laboratory conditions at 28±2 °C and 60±10 % RH at an 8L:16D photoperiod. The dried materials were partially ground using a domestic blender (Preethi model 7540; Preethi Kitchen Appliances Pvt. Ltd., Chennai) and transferred into each of four plastic jars of 1 L capacity, and stored at room temperature for further studies.

Extraction of essential oil

The essential oils were obtained by steam distillation from the air-dried stem and root samples (100 g each) of *T. purpurea* and *I. carnea* using a Clevenger apparatus for 6 h. The volatile distillate was collected. The distillate was saturated with sodium chloride solution and added to 0.5–1.0 mL diethyl ether. Then, the ether layer and aqueous layer were separated and the organic layer was dried over anhydrous sodium sulfate to remove the moisture and the ether was distilled off over a water bath maintained at 50 °C. The remainder, *i.e.*, oils, were dried over anhydrous sodium sulfate, weighed and stored at 5 °C for further analysis.

Gas Chromatography–Mass Spectrometry (GC–MS) analyses

Essential oils of *T. purpurea* and *I. carnea* were subjected to gas chromatography (Hewlett-Packard gas chromatography model 5890) equipped with megabore column (10 m×0.53 mm i.d., fused silica) packed with OV-1 and a nitrogen phosphorous detector (NPD). The temperature program used for the analysis was as follows: initial temperature at 80 °C, held for 2 min, increased to 120 °C at 28 °C min^{-1}, held isothermal at 120 °C for 1 min, increase to 200 °C at 58 °C min^{-1}, and held isothermal at 200 °C for 2 min; injection temperature, 220 °C; detector temperature, 250 °C; and nitrogen (1.0 mL min^{-1}) was the carrier gas. The samples (0.1 % in absolute ethanol) were injected into the GC by split mode with a split ratio of 1/20. Methyl salicylate was used as an internal standard. The percentages of the constituents were calculated by electronic integration of FID peak areas without response factor correction. GC–MS analyses were performed using JEOL JMS-D300 mass spectrophotometer at 70 eV using electron impact ionization with the source at ambient temperature. The conditions were same as those described above. Injector temperature, 250 °C; split flow, 50 mL min^{-1}; carrier gas, He (1.0 mL min^{-1}). Diluted sample (1.0 mL, 1:10 in Et_2O) was injected manually. MS: at 70 eV; mass range 35–425 amu; ion source temperature, 200 °C; interface temperature, 250 °C. The chemical constituents were identified based on the comparison of their mass spectral pattern and retention indices with those obtained from the Wiley 138.L, NBS 75K.L, and SDBS libraries, as well as those published by Adams.[32] The retention indices (*RI*) were calculated according to the literature.[33]

Estimation of percentage of essential oil

Five gram coarsely powdered *T. purpurea* and *I. carnea* stem and root were extracted with petroleum ether in a Soxhlet apparatus for about 1 h. The thimble containing unextracted

portion was taken out, dried and weighed. The percentage of oil present in the material was calculated.[34]

Extraction and estimation of total lipids

One gram of partially powdered plant material was mixed with 10 mL mixture of diethyl ether and ethanol (3:1) in a mortar and centrifuged at 2000 rpm for 10 min. The supernatant was transferred into a separating funnel and 2 mL of 0.05 M potassium chloride solution added. The mixture was allowed to stand until two layers separated. The lipids were present in organic layer. The salt prevented any emulsion formation.[34]

Insect rearing

O. longicollis adults (Fig. 1) were obtained from a colony maintained in the Crop Protection Research Centre, St. Xavier's College (Autonomous), Palayamkottai, Tamil Nadu, India. The insects were reared on fresh banana pseudostem. Cultures were maintained in the dark at 28±1 °C and 70±5 % relative humidity.

Fig. 1. Banana pseudostem weevil *Odoiporus longicollis* adult.

Repellent activity bioassay

Bioassays were conducted according to the method of Sahayaraj and Kombiah.[35] A Whatman number 1 paper strip of 1.5 cm×1.5 cm was selected for the determination of the repellent activity. A Y-shaped olfactometer[35] was used to conduct two-choice bioassays. A piece of paper soaked in 1 mL of essential oil was placed in the experimental arm, whereas the control arm contained a piece of paper soaked in the same amount of solvent, diethyl ether and ethanol in 3:1 ratio (negative control) only. Three one-week old adult male beetles were introduced into the non-reacting base arm of the olfactometer. The numbers of insects in each arm were counted after 30, 60, 90, 120, 150 and 180 min. The insects chose either the test chamber or control chamber or neither. Insects that moved towards the solvent showed attractant phenomena while those moving towards the EOs showed a repellency effect. If the insect chose neither of the chambers, then it was considered that the insect made no choice. The experiment was replicated six times with different insects of the same age group. A similar protocol was followed for one-week old adult female beetles.

Statistical analysis

The weevils preferred either fresh EOs, solvent or neither. Weevil choosing the EOs was considered as attractant. If the weevil chose neither of the chambers then it was considered that weevil made no choice. From the recorded observations, the Access Proportion Index (*API*) was calculated using the formula:[35]

$$API = NS–NC/NS+NC$$

where *NS* = number of insects choosing the sample (EO) side and NC = number of insects choosing the control (solvent) side.

The approaching behavior of the insect between EOs and solvent was subjected to the χ^2 test and the significance was expressed at 5 % level using MS-Excel 2007. The same package was used to analyze the approaching data of male and female beetles.

RESULTS AND DISCUSSION

Total lipid and oil contents

More total lipid (4.8 mL g^{-1} dry weight stem) and content of oil (12.53 mL g^{-1} dry weight stem) was found in the stem of *T. purpurea* than in root (4.0 mL g^{-1} dry weight root and 8.0 % for total lipid and oil, respectively). Similarly, a higher oil (13.75 % oil) and total lipid content (4.5 mL g^{-1} dry weight stem) was observed in the stem of *I. carnea* than in the root (4.0 ml g^{-1} dry weight root and 5.75 % of total lipid and oil, respectively). However, *I. carnea* yielded only 0.15 to 0.2 % of oil by the steam distillation method.[36] In general, the total lipid content as well as biosynthesis of essential oil was higher in the stem than in the root of flowering plants, depending on the metabolic state and preset developmental differentiation program of the synthesizing tissue.[37]

Chemical composition of essential oil

The steam-distillation of *T. purpurea* and *I. carnea* stem and root yielded yellowish and slightly viscous essential oils. The EOs of *T. purpurea* and *I. carnea* showed distinct unpleasant odors. This might be due to the presence of caryophyllene, geraniol, linalool, eugenol,[38] linalool, methyl chavicol, 1,8-cineole, *α*-pinene, *β*-pinene, myrcene, ocimene, terpinolene, camphor, terpinen-4-ol, *α*-terpineol, eugenol and sesquiterpenes.[39]

In all, 9 and 10 compounds were identified by gas chromatography and mass spectrometry data in the essential oils of stem and root of *T. purpurea* and *I. carnea*, respectively (Tables I and II). Of these, three volatile compounds were

TABLE I. Chemical constituents of the essential oils from *T. purpurea* stem and root analyzed by GC–MS; *RT*: retention time relative to *n*-alkanes (C_6–C_{24}) on an RTX-5 MS column

Plant part	Compound name	Molecular formula	*RT* / min	Content, %
Stem	*β*-Caryophyllene oxide	$C_{15}H_{24}O$	21.69	3.53
	Bulnesol	$C_{15}H_{26}O$	22.10	3.30
	Tetradecanoic acid	$C_{14}H_{28}O_2$	22.32	2.48
	Caryophyllene oxide	$C_{15}H_{24}O$	22.69	10.03
	Hexadecanoic acid	$C_{16}H_{32}O_2$	25.62	69.61
	Linoleic Acid	$C_{18}H_{32}O_2$	27.29	11.05
Root	Isolongifolan-8-ol	$C_{15}H_{24}$	20.45	5.74
	Epiglobulol	$C_{15}H_{26}O$	20.98	12.66
	Azulene	$C_{10}H_8$	22.10	10.28
	Bulnesol	$C_{15}H_{26}O$	22.85	16.82
	Hexadecanoic acid	$C_{16}H_{32}O_2$	25.63	46.97
	Linoleic acid	$C_{18}H_{32}O_2$	27.29	7.52

common in both the stem and root of the plants. However, *Tephrosia egregia* Sandw. stem oil consisted of 10 volatile compounds.[40] The essential oils mainly contained hexadecanoic acid with *α*-pinene, *γ*-terpinene, *β*-pinene, isoamyl bromide, *p*-cymene, limonene and geraniol being minor components.

TABLE II. Chemical constituents of the essential oils from *I. carnea* stem and root analyzed by GC–MS; *RT*: retention time relative to *n*-alkanes (C_6–C_{24}) on an RTX-5 MS column

Plant part	Compound name	Molecular formula	*RT* / min	Content, %
Stem	2-(12-Pentadecynyloxy)tetrahydro-2*H*-pyran	$C_{20}H_{36}O_2$	23.82	2.84
	1-Octadecanol	$C_{18}H_{32}O$	26.82	3.22
	Hexadecanoic acid	$C_{16}H_{32}O_2$	25.64	70.61
	Epiglobulol	$C_{15}H_{26}O$	22.52	4.35
	Squalene	$C_{30}H_{50}$	28.16	3.00
	1-Octadecanol	$C_{18}H_{38}O$	27.29	15.99
Root	2-Ethyl-1,3-dimethylbenzene	$C_{10}H_{14}$	10.11	1.52
	2-(12-Pentadecynyloxy)tetrahydro--2*H*-pyran	$C_{20}H_{36}O_2$	23.82	1.70
	3-Furanyl[2-hydroxy-4-methyl-2--(2-methylpropyl)cyclopentyl]-methanone	$C_{15}H_{22}O_3$	24.47	1.68
	2,2-Dideuterooctadecanal	$C_{18}H_{36}O$	24.80	2.46
	Hexadecanoic acid	$C_{16}H_{32}O_2$	25.64	88.89
	Linoleic acid	$C_{18}H_{32}O_2$	27.28	3.76

To the best of our knowledge, this is the first report regarding the presence of the chemical constituents in the oils of stem and root of *T. purpurea* and *I. carnea.* Six compounds each were identified in stem and root of *T. purpurea*. Hexadecanoic acid was the main compound of the stem (69.61 %) and root (46.97 %) of *T. purpurea.* Other common components in the oil of *T. purpurea* were linoleic acid, bulnesol and epiglobulol. Similarly, in *I. carnea*, the fatty acid (hexadecanoic acid) was found to be the principal and common constituent of the root (88.89 %) and stem (70.61 %) oils. Other minor chemical components in the oil of *I. carnea* were epiglobulol, 1-octadecanol, squalene and 2-(12-pentadecynyloxy)-tetrahydro-2*H*-pyran in the stem and linoleic acid, 2,2-dideuteroctadecanal, 2-(12-pentadecynyloxy)-tetrahydro-2*H*-pyran, 3-furanyl[2-hydroxy-4--methyl-2-(2-methylpropyl)-cyclopentyl]methanone and 2-ethyl-1,3-dimethylbenzene in the root. Chemo-profiling of these oils showed differences in the composition among the studied species although volatile constituents were previously reported in other species of the *Tephrosia* genus.[40,41] Caryophyllene oxide was the major component (63.9 %) of leaves of *T. cinerea* Pers,[41] whereas the major components of the stem oil of *T. egregia* Sandw. were geijerene and pregeijerene.[40] *T. vogelii* Hook f. seed consisted more hexadecanoic acid (pal-

mitic acid, 18.70 %) along with tetradecanoic acid, pentadecanoic acid, heptadecanoic acid, stearic acid, oleic acid, linoleic acid, linolenic acid, eicosanoic acid, heneicosanoic acid, docosanoic acid, tricosanoic acid and tetracosanoic acid.[42]

Repellent activity

T. purpurea stem oil showed pronounced repellency (overall mean *API* = = –0.73) (df_1, p = 3.94, p < 0.005) of *O. longicollis* adults followed by *I. carnea* stem oil (overall mean *API* = –0.70) (df_1, p = 3.86, p < 0.05, Table III). Invariably, the tested oils deterred the male adults of *O. longicollis* more than the female adults (t = 1.743; p < 0.05). Similarly, both neem oil and pongamia oil showed strong repellent effects on *O. longicollis*.[43] The results showed that repellent activity of the oil might be due to the presence of hexadecanoic acid or the mixture of this with the other chemical constituents of the EOs that are toxic to the insect and are present in the oil. The principlal compound, hexadecanoic acid possesses a variety of biological activities including insecticidal activity.[44–46]

TABLE III. Repellent activity of *T. purpurea* and *I. carnea* oils on pseudostem weevil *Odoiporus longicollis*; *t* test performed between male and female of the same category; the same letter means insignificant at the 5 % level

Plant	Part Used	Insect sex	Time after the introduction of insect, min						Mean repellence
			30	60	90	120	150	180	
T. purpurea	Stem	Male	–0.64[b]	–0.69[b]	–0.75[b]	–0.79[b]	–0.80[b]	–0.86[b]	–0.73[b]
		Female	–0.52[a]	–0.63[a]	–0.67[a]	–0.67[a]	–0.70[a]	–0.72[a]	–0.63[a]
	Root	Male	–0.16[b]	–0.33[b]	–0.50[a]	–0.62[b]	–0.63[b]	–1.0[b]	–0.33[b]
		Female	–0.09[a]	–0.15[a]	–0.20[a]	–0.27[a]	–0.57[a]	–0.76[a]	–0.38[a]
I. carnea	Stem	Male	–0.33[b]	–0.55[b]	–0.67[b]	–0.78[b]	–0.81[b]	–1.0[b]	–0.70[b]
		Female	–0.14[a]	–0.37[a]	–0.55[a]	–0.75[a]	–0.80[a]	–0.89[a]	–0.59[a]
	Root	Male	–0.28[b]	–0.40[b]	–0.53[b]	–0.77[a]	–0.91[b]	–1.0[b]	–0.69[b]
		Female	–0.25[a]	–0.30[a]	–0.33[a]	–0.74[a]	–0.80[a]	–0.82[a]	–0.56[a]

To the best of our knowledge, this is the first report on the repellent action of the essential oils from the root and stem of these plants against any pests, particularly *O. longicollis*. Furthermore, this fatty acid should be separated during bio-oil production and utilized as a source of an effective insect repellent compound. The essential oils of the stem (70 %) and root (89 %) of *T. purpurea* and *I. carnea* contained high levels of hexadecanoic acid, which could be utilized as an insect repellent. The development and utilization of this natural pesticide would also help to decrease the negative impact of synthetic chemicals, such as residues, resistance and environmental pollution.

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И З В О Д

ХЕМИЈСКИ САСТАВ ЕТАРСКИХ УЉА *Tephrosia purpurea* И *Ipomoea carnea* И ЊИХОВА РЕПЕЛЕНТНА АКТИВНОСТ СПРАМ ЖИШКА *Odoiporus longicollis*

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Хемијски састав етарских уља добијених из стабла и корена биљака *Tephrosia purpurea* (Linn.) Pers. и *Ipomoea carnea* Jacq. је анализиран гасно–масеном спектрометријом (GC–MS). Укупни удео липида и уља је био висок у свим узорцима. Етарско уље из стабла и корена *T. purpurea* је садржало 9 састојака, а из *I. carnea* 8. Хексадеканска киселина је била главни састојак у свим узорцима: у стаблу и корену *T. purpurea* је чинила 69,61 и 46,97 %, а код *T. purpurea* 70,61, односно 88,89 %. Налази ове студије указују да би се етарска уља *T. purpurea* и *I. carnea* могла користити као извор хексадеканске киселине за индустријске сврхе. Етарска уља су испољила јачу репелентну активност спрам мужјака (–0,73 и –0,70, уља из стабала *T. purpurea* и *I. carnea*) него женки (–0,63 и –0,59) жишка банане, *Odoiporus longicollis*. Резултати су показали да активни састојци етарских уља стабла *T. purpurea* и *I. carnea* могу наћи примену као природни репеленти за контролу жишка *O. longicollis*.

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