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Full Length Research Paper

Chemical composition and toxicities of essential oil of *Illicium fragesii* fruits against *Sitophilus zeamais*

Cheng Fang Wang[1], Peng Liu[2], Kai Yang[3], Yan Zeng[1], Zhi Long Liu[3]*, Shu Shan Du[1]* and Zhi Wei Deng[2]

[1]State Key Laboratory of Earth Surface Processes and Resource Ecology, Beijing Normal University, Haidian District, Beijing 100875, P. R. China.
[2]Analytic and Testing Center, Beijing Normal University, Haidian District, Beijing 100875, P. R. China.
[3]Department of Entomology, China Agricultural University, 2 Yuanmingyuan West Road, Haidian District, Beijing 100193, P. R. China.

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The aim of this research was to determine the chemical composition and toxicities of essential oil derived from *Illicium fargesii* Finet et Gagnep fruits against the maize weevil (*Sitophilus zeamais* Motsch). Essential oil of *I. fargesii* fruits was obtained from hydrodistillation and was investigated by GC (Gas Chromatography) and GC–Mass Spectrometry (GC-MS). A total of 43 components of the essential oil of *I. fargesii* fruits were identified. The principal compounds in *I. fargesii* essential oil were α-terpineol (11.4%), carvone (10.9%), d-limonene (9.8%), trans-carveol (6.6%) and trans-pinocarveol (5.5%). The essential oil of *I. fargesii* possessed strong fumigant toxicity against the maize weevil with an LC_{50} value of 11.36 mg/L air. The essential oil of *I. fargesii* also exhibited contact toxicity against *S. zeamais* adults with an LD_{50} value of 28.95 µg/adult. The result indicate that the essential oil of *I. fargesii* show potential in terms of fumigant and contact toxicities against grain storage insects.

Key words: *Illicium fargesii*, *Sitophilus zeamais*, fumigant, contact toxicity, essential oil composition.

INTRODUCTION

Illicium fargesii Finet et Gagnep (Family: Illiciaceae), a medium-sized flowering tree, is indigenous to southwestern China and its fruit is used locally as a folk medicine for the treatment of rheumatism (Moriyama et al., 2008). It is one of adulterants of the commonly used non-toxic spice, Chinese star anise, *I. verum* (Jiangsu New Medical College, 1977). Several sesquiterpenoids, prenylated phenylpropanoids, and sesqui-neolignans have been isolated from this plant (Moriyama et al., 2007, 2008). During our mass screening program for new agrochemicals from the wild plants, essential oil of *I. fargesii* fruits was found to possess insecticidal activities against the maize weevil, *Sitophilus zeamais* (Motschulsky). A literature survey has shown that there is no report on the volatile constituents and insecticidal activity of *I. fargesii*, thus we decided to investigate the chemical constituents and insecticidal activities of the essential oil of *I. fargesii* fruits against insects for the first time.

The maize weevil, *S. zeamais* is one of the most widespread and destructive primary insect pests of stored cereals (Liu and Ho, 1999). Infestations not only cause significant losses due to the consumption of grains; they also result in elevated temperature and moisture conditions that lead to an accelerated growth of molds, including toxigenic species (Magan et al., 2003). The conventional control of the stored product insects has been with the use of synthetic insecticides, either directly applied to grains or by gas fumigation. Fumigation plays a very important role in insect pest elimination in stored products not only because of their ability to kill a broad spectrum of pests but because of their easy penetration into the commodity while leaving minimal residues (Zettler and Arthur, 2000). The currently used synthetic fumigant is still the most effective means for the

*Corresponding author.s E-mail: zhilongliu@cau.edu.cn; dushushan@bnu.edu.cn. Tel/Fax: 86-10-62732800.

protection of stored food, feedstuffs and other agricultural commodities from insect infestation. However, repeated use of those synthetic fumigants for decades has disrupted biological control by natural enemies and led to resurgence of stored-product insect pests, sometimes resulted in the development of resistance, and had undesirable effects on non-target organisms (Isman, 2000). Moreover, the use of methyl bromide will be prohibited in the near future because of its ozone depletion potential (Anonymous, 1993). These problems have highlighted the need to develop new types of selective insect-control alternatives with fumigant action. Plant essential oils and their components have been shown to possess potential to be developed as new fumigants and they may have the advantage over conventional fumigants in terms of low mammalian toxicity, rapid degradation and local availability (Isman, 2000, 2006). Essential oils derived from many plant species have been evaluated for fumigant toxicity against stored product insects so far (Rajendran and Srianjini, 2008).

MATERIALS AND METHODS

Insects

S. zeamais were obtained from the laboratory cultures maintained for the last ten years in the dark in incubators at 27 to 29°C and 70 to 80% relative humidity. *S. zeamais* adults were reared on whole wheat at 12 to 13% moisture content. Unsexed adults of the maize weevils used in all the experiments were about two weeks old.

Plant material

5 kg of fruits of *I. fargesii* were collected in August 2010 from Dali City (25.34° N latitude and 100.13° E longitude), Yunnan Province, China. The species was identified by Dr. Liu, Q.R., College of Life Sciences, Beijing Normal University, Beijing 100875, and the voucher specimens (BNU-DuShushan-Illi-10-345) were deposited at the Herbarium (BNU) of College of Life Sciences, Beijing Normal University. The fruits were air-dried and were first ground to powder using a grinding mill (Retsch Muhle, Germany). Each 600 g portion of the powder ground was mixed in 1,800 ml of distilled water and soaked for 3 h. The mixture was then boiled in a round-bottom flask, and distilled for 6 to 8 h. Essential oil from distillation was collected in a flask. Separation of the essential oil from the aqueous layer was done in a separatory funnel, using the non-polar solvent, *n*-hexane. The solvent was evaporated using a vacuum rotary evaporator (BUCHI Rotavapor R-124, Switzerland). The sample was dried over anhydrous Na_2SO_4 and kept in a refrigerator (4°C) for subsequent experiments.

Analysis of the essential oils

Components of the essential oil were separated and identified by gas chromatography-flame ionization detection (GC-FID) and gas chromatography–mass spectrometry (GC–MS) Agilent 6890N gas chromatography hooked to Agilent 5973N mass selective detector. The same column and analysis conditions were used for both GC and GC/MS. They were equipped with a flame ionization detector and capillary column with HP-5MS (30 m × 0.25 mm × 0.25 μm).

The GC settings were as follows: the initial oven temperature was held at 60°C for 1 min and ramped at 10°C min^{-1} to 180°C for 1 min, and then ramped at 20°C min^{-1} to 280°C for 15 min. The injector temperature was maintained at 270°C. The samples (1 μl) were injected neat, with a split ratio of 1: 10. The carrier gas was helium at flow rate of 1.0 ml min^{-1}. Spectra were scanned from 20 to 550 m/z at 2 scans s^{-1}. Most constituents were identified by gas chromatography by comparison of their retention indices with those of the literature or with those of authentic compounds available in our laboratories. The retention indices were determined in relation to a homologous series of *n*-alkanes (C_8 to C_{24}) under the same operating conditions. Further identification was made by comparison of their mass spectra with those stored in NIST 05 (Standard Reference Data, Gaithersburg, MD 20899, USA) and Wiley 275 libraries (Wiley, New York) or with mass spectra from literature (Adams, 2007). Component relative percentages were calculated based on GC peak areas without using correction factors.

Fumigant toxicity

Range-finding studies were run to determine the appropriate testing concentrations of *I. fargesii* essential oil. A Whatman filter paper (diameter 2.0 cm, CAT No. 1001020) was placed on the underside of the screw cap of a glass vial (diameter 2.5 cm, height 5.5 cm, volume 24 ml). 10 μl of 2.63 to 20.00% (v: v, 6 concentrations) of the essential oil of *I. fargesii* fruits was added to the filter paper. The solvent was allowed to evaporate for 15 s before the cap was placed tightly on the glass vial (with 10 unsexed insects) to form a sealed chamber. Preliminary experiments demonstrated that 15 s were sufficient for the evaporation of solvents. Fluon (ICI America Inc) was used inside glass vial to prevent insects from the treated filter paper. *n*-Hexane was used as controls. Six replicates were used in all treatments and controls and they were incubated at 27 to 29°C and 70 to 80% relative humidity for 24 h. Mortality of insects was observed and results from all replicates were subjected to probit analysis using the PriProbit Program V1.6.3 to determine LC_{50} values (Sakuma, 1998).

Contact toxicity using topical application

The contact toxicity of the essential oil of *I. fargesii* fruits against *S. zeamais* adults was measured as described by Liu and Ho (1999). Range-finding studies were run to determine the appropriate testing concentrations of the essential oil. A serial dilution of the essential oil of *I. fargesii* (2.69 to 10.00%; 6 concentrations) was prepared in *n*-hexane. Aliquots of 0.5 μl of the dilutions were applied topically to the dorsal thorax of the insects. Controls were determined using *n*-hexane. Six replicates were used in all treatments and controls. Both treated and control insects were then transferred to glass vials (10 insects/vial) with culture media and kept in incubators at 27 to 29°C, and 70 to 80% R.H. Mortality of insects was observed after 24 h and the experiments were repeated in three times. The LD_{50} values were calculated by using Probit analysis (Sakuma, 1998). Positive control, pyrethrum extract (25% pyrethrine I and pyrethrine II) was purchased from Fluka Chemie.

RESULTS AND DISCUSSION

Essential oil

The steam distillation for 6 h of fruits of *I. fargesii* afforded essential oil (yellow) with a yield of 0.96% (v/w) and the density of the concentrated essential oil was determined

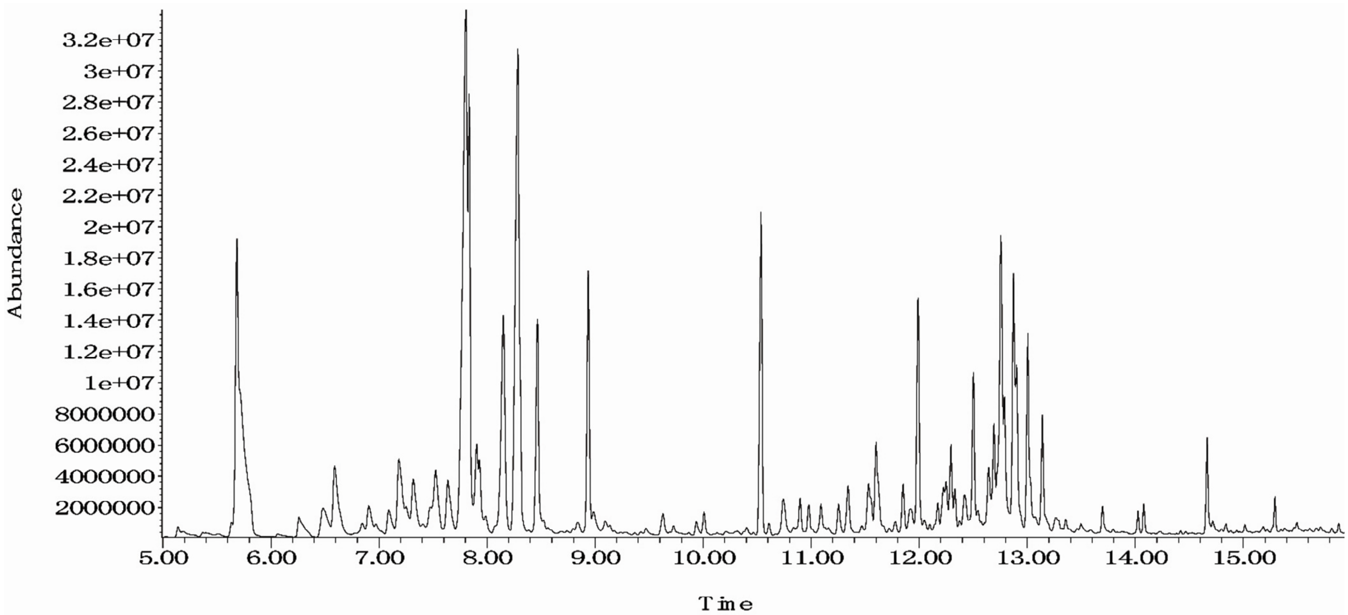

Figure 1. Total ion chromatogram of *Illicium fargesii* essential oil.

as 0.88 g/ml. The GC and GC–MS analysis of the essential oil of *I. fargesii* fruits led to the identification and quantification of a total of 43 components accounting for 95.7% of the total components present (Figure 1). α-Terpineol (11.4%), carvone (10.9%), d-limonene (9.8%), trans-carveol (6.6%), and trans-pinocarveol (5.5%) are the main components of the essential oil of *I. fargesii* followed by copaene (4.2%), caryophyllene (4.1%), and γ-eudesmol (4.0%) (Table 1). Monoterpenoids (included 12.0% monoterpene hydrocarbons and 48.6% oxygenated monoterpenes) represented 23 of the 43 compounds, corresponding to 60.3% of the whole oil while only 19 of the 43 constituents were sesquiterpenoids (included 21% sesquiterpene hydrocarbons and 11.2% oxygenated sesquiterpenes), corresponding to 32.2% of the crude essential oil. The chemical composition of the essential oil of *I. fargesii* fruits was reported here for the first time.

Insecticidal activities

The essential oil of *I. fargesii* fruits shows contact toxicity against *S. zeamais* adults with an LD_{50} value of 28.95 μg/adult (Table 2). However the essential oil demonstrated six times less acute toxicity against the weevil than the positive control (pyrethrum extract, 25% pyrethrine I and pyrethrine II) because the pyrethrum extract exhibited acute toxicity to the maize weevils with an LD_{50} value of 4.29 μg/adult (Table 2). The essential oil of *I. fargesii* fruits also exhibited strong fumigant activity against *S. zeamais* adults with an LC_{50} value of 11.36 mg/L air (Table 2). The currently used grain fumigants, methyl bromide (MeBr) and phosphine were reported to have fumigant activity (24 h) against *S. zeamais* adults with LC_{50} values of 0.67 mg/L and 6 μg/L air, respectively (Liu and Ho, 1999). The essential oil of *I. fargesii* fruits was 17 times less toxic to the maize weevil compared with the commercial fumigant MeBr. However, compared with the other essential oils in the literature, the essential oil of *I. fargesii* fruits possessed the same level or stronger fumigant toxicity against *S. zeamais* adults, such as essential oils of *Murraya exotica* (LC_{50} = 8.29 mg/L) (Li et al., 2010), *Schizonpeta multifida* (LC_{50} = 8.33 mg/L) (Liu et al., 2011a), *Artemisia lavandulaefolia* (LC_{50} = 11.2 mg/L) and *A. sieversiana* (LC_{50} = 15.0 mg/L) (Liu et al., 2010), *A. vestita* (LC_{50} = 13.42 mg/L) (Chu et al., 2010a), *Illicium simonsii* (LC_{50} = 14.95 mg/L) (Chu et al., 2010b), and *Ostericum sieboldii* (LC_{50} = 27.39 mg/L) (Liu et al., 2011b). The aforementioned findings suggest that fumigant activity of the essential oil of *I. fargesii* fruits is quite promising by considering that the currently used fumigants are synthetic insecticides and it shows potential to be developed as a possible natural fumigant for the control of stored product insects. However, for the practical application of the essential oil as novel fumigant, further studies on the safety of the essential oil to humans and on development of formulations are necessary to improve the efficacy and stability and to reduce cost.

Several essential oils from Genus *Illicium* have been evaluated for their fumigant and contact toxicities against insect pests such as stored product insects (Kim et al., 2003; Chaubey, 2008; Chu et al., 2010b), termites (Park and Shin, 2005) and some show potential in the development of new natural insecticides. Moreover, (*E*)-

Table 1. Constituents identified from the essential oil of *Illicium fargesii* fruits. ρ

Compound	**RI***	**Peak area (%)**
α -Pinene	939	0.1
Sabinene	973	0.1
β -Pinene	982	0.4
d-Limonene	1031	9.8
γ-Terpinene	1057	0.1
ρ-Cymenene	1086	1.1
α-Fenchol	1104	0.2
Hotrienol	1108	1.3
ρ-Mentha-1,5,8-triene	1113	0.2
trans- ρ-Mentha-2,8-dien-1-ol	1123	0.7
cis- ρ-Mentha-2,8-dien-1-ol	1128	0.6
Cosmene	1130	0.2
trans-Pinocarveol	1138	5.5
(-)-Borneol	1160	1.8
4-Terpineol	1178	1.2
α-Terpineol	1189	11.4
Myrtenol	1196	1.1
trans-Carveol	1216	6.6
cis-Carveol	1226	0.7
cis-1(7),8- ρ-Menthadien-2-ol	1231	3.3
Carvone	1248	10.9
Geranial	1267	2.1
(+)-Bornyl acetate	1287	3.5
1,2-Dihydro-1,1,6-trimethylnaphthalene	1355	2.7
Copaene	1374	4.2
Geranyl acetate	1379	0.2
cis-α-Bergamotene	1413	0.2
Caryophyllene	1420	4.1
β-Aromadendrene	1446	0.8
1,5,9,9-tetramethyl-, *Z*, *Z*, *Z*-1,4,7,-Cycloundecatriene	1454	0.6
Neryl propanoate	1455	0.2
(-)-Alloaromadendrene	1458	0.4
α-Amorphene	1482	1.5
cis-β-Guaiene	1490	0.9
(-)-α-Selinene	1494	3.1
α-Muurolene		0.7
(-)-β-Cadinene	1518	1.9
Caryophyllenyl alcohol	1575	1.1
(-)-Spathulenol		0.9
Caryophyllene oxide		2.0
γ-Eudesmol	1631	4.0
τ-Cadinol	1642	1.4
α-Cadinol	1653	1.9
Total identified		95.7
Monoterpene hydrocarbons		12.0
Oxygenated monoterpenes		48.6
Sesquiterpene hydrocarbons		21.0
Oxygenated sesquiterpenes		11.2
Other		2.9

*RI, retention index as determined on a HP-5MS column using the homologous series of *n*-hydrocarbons.

Table 2. Toxicities of *Illicium fargesii* essential oil against *Sitophilus zeamais* adults.

Toxicity	Treatment	LD_{50} /LC_{50}*	95% FL	Slope ± SE	Chi square (X^2)
Contact	*Illicium fargesii*	28.95	27.23 - 30.43	5.59 ± 0.58	17.08
	Pyrethrum extract	4.29	3.86 - 4.72	0.72 ± 0.01	13.51
Fumigant	*Illicium fargesii*	11.36	10.78 - 12.68	3.62 ± 0.36	22.96
	MeBr	0.67[a]	-	-	-

*Contact toxicity: LD_{50} =µg/adult; Fumigant: LC_{50} = mg/L air; [a]Liu and Ho (1999).

anethole, active component from the essential oil of Chinese star anise, *I. verum* against German cockroaches was isolated and identified (Chang and Ahn, 2002). However, there is no report on the volatile constituents and toxicities of *I. fargesii* against insects; we reported here contact and fumigant toxicities of the essential oil of *I. fargesii* fruits against insects and chemical composition of the essential oil for the first time.

In previous studies, these main constituents of the essential oil had been demonstrated to possess toxicity against insects and mites. For example, α-terpineol exhibited strong contact and fumigant toxicities against several insects and mites [*Blattella germanica* (Jang et al., 2005), *Lipaphis pseudobrassicae* (Sampson et al., 2005), *Pediculus humanus capitis* (Yang et al., 2009) and the two-spotted spider mite, *Tetranychus urticae* (Lee et al., 1997)]. The possible modes of action of α-terpineol suggested are interference with the octopaminergic system of insects (Enan, 2001). Another main compound, carvone also possessed insecticidal activity against several species of stored product insects (Sanchez-Ramos and Castanera, 2001; Abdelgaleil et al., 2009; Lopez et al., 2010; Fang et al., 2010) and the two-spotted spider mite, *T. urticae* (Badawy et al., 2010). d-Limonene has been commercialized for use as flea dips and shampoos for pets as well as sprays and aerosols (Ibrahim et al., 2001). It has been demonstrated to possess insecticidal activity against several stored-product insects (Tripathi et al., 2003; Fang et al. 2010). These compounds were demonstrated to be a potent inhibitor of acetylcholinesterase (AChE) activity from larvae of several stored product insects (Abdelgaleil et al., 2009; Lopez et al., 2010). The aforementioned findings suggest that the isolation and identification of the bioactive compounds in the essential oil of *I. fargesii* fruits are of utmost importance so that their potential application in controlling stored-product pests can be fully exploited.

The fruits of *I. fargesii* were used as a folk medicine for the treatment of rheumatism (Moriyama et al., 2008) and sometimes were used as one of adulterants of Chinese star anise. However, there are no toxicity data for *I. fargesii* fruits available on human consumption. Therefore, any attempt to develop an *I. fargesii* essential oil-derived agrochemical must be carefully evaluated for harmful effects.

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