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Chemical Compositions of the Leaf Essential Oils of *Aralia spinosa* from Three Habitats in Northern Alabama

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ABSTRACT

Aralia spinosa leaves were collected from three different habitats in north Alabama. The leaf essential oils were collected by hydrodistillation and analyzed by gas chromatography/mass spectrometry (*GC-MS*). *The most abundant components of A. spinosa essential oils were the sesquiterpenes germacrene D* (28.0% - 37.3%), (*E*)-*caryophyllene* (8.2% - 15.7%), *and α-humulene* (1.9% - 4.9%); *the monoterpene myrcene* (*up to* 15.1%), *and the fatty-acid-derivative* (2*E*)-*hexenal* (*trace to* 28.9%). *Fatty-acid derivatives and monoterpene hydrocarbons were more abundant in samples from suburban Huntsville than those from "natural" habitats* (*Monte Sano Mountain*, *Wheeler National Wildlife Refuge*), *while sesquiterpene hydrocarbons were more abundant in the natural/wild samples*.

Keywords: *Aralia Spinosa*, *Araliaceae*, *Leaf Essential Oil*, *Chemical Composition*, *Germacrene D*, *Myrcene*, (*E*)-*Caryophyllene*, (2*E*)-*Hexenal*

1. Introduction

Aralia spinosa L. (Araliaceae), "devil's walking stick", is a large shrub/medium-sized (up to 10 m tall) tree native to the southeastern United States. The leaves are very large (up to 1.5 m long, 1 m wide), bipinnate with ovate leaflets, 2 cm - 10 cm long. The plant produces numerous tiny white flowers in umbels (July-September) and bluish drupes [1]. To our knowledge, the leaf essential oil of *A. spinosa* has not been previously examined. In this work, we present and compare the chemical compositions of the leaf essential oils of *A. spinosa* from three habitats in north Alabama.

2. Materials and Methods

2.1. Plant Material

Leaves of *A. spinosa* were collected (July, 2010) from four mature trees growing in north Alabama: two trees from suburban Huntsville city (34˚38"77'N, 86˚33"45'W, 187 m elevation), one tree from Monte Sano Mountain (34˚44"85′N, 86˚31"77'W, 427 m elevation), and one tree from Wheeler National Wildlife Refuge (34˚37"40'N, 86˚57"08'W, 122 m elevation). The plants were identified and collected by P. Davé, and a voucher specimen has been deposited in the University of Alabama in Huntsville herbarium. The freshly chopped leaves from each plant were hydrodistilled using a Likens-Nickerson apparatus. Continuous extraction of the distillates with CH_2Cl_2 for three hours gave clear colorless essential oils (Huntsville #1, 9.52%; Huntsville #2, 6.18%; Monte Sano, 1.05%; Wheeler NWR, 1.07%).

2.2. Gas Chromatography—Mass Spectrometry

The leaf essential oils of *A. spinosa* were subjected to gas chromatographic-mass spectral analysis on an Agilent system consisting of a Model 6890 gas chromatograph, a Model 5973 mass selective detector [MSD, operated in the EI mode (electron energy = 70 eV), scan range = 45 amu - 400 amu, and scan rate = 3.99 scans/sec], and an Agilent ChemStation data system. The GC column was an HP-5ms fused silica capillary with a (5% phenyl)-polymethylsiloxane stationary phase, film thickness of 0.25 μm, a length of 30 m, and an internal diameter of 0.25 mm. The carrier gas was helium with a column head pressure of 48.7 kPa and a flow rate of 1.0 mL/min. Inlet temperature was 200˚C and interface temperature was 280˚C. The GC oven temperature program was used as follows: 40˚C initial temperature, hold for 10 mins; in-

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creased at 3˚C/min to 200˚C; increased at 2˚C/min to 220˚C. A 1% w/v solution of the sample in CH_2Cl_2 was prepared and 1 μL was injected using a splitless injection technique. Identification of the oil components was based on their retention indices determined by reference to a homologous series of *n*-alkanes, and by comparison of their mass spectral fragmentation patterns with those reported in the literature [2] and stored on the MS library [NIST database (G1036A, revision D.01.00)/ChemStation data system (G1701CA, version C.00.01.080)]. The percentages of each component are reported as raw percentages based on total ion current without standardization. The leaf essential oil compositions of *A*. *spinosa* are summarized in **Table 1**.

Table 1. Chemical compositions of *Aralia spinosa* leaf essential oils.

RI	Compound	Percent Composition			
		Suburban Huntsville		Monte Sano	Wheeler
		#1	#2	Mountain	NWR
760	(2*E*)-Pentenal	0.1	0.4	0.2	---
773	(2*Z*)-Pentenol	1.7	2.0	0.8	---
795	(3*Z*)-Hexenal	---[a]	8.3	---	---
800	Hexanal	2.8	---	1.6	tr[b]
833	Furfural	---	---	0.2	---
845	Unidentified hexenal	---	---	0.5	---
854	(2*E*)-Hexenal	24.7	28.9	13.8	tr
911	(2*E*,4*E*)-Hexadienal	0.2	0.6	tr	---
934	*α*-Thujene	---	---	tr	---
935	*α*-Pinene	tr	0.6	1.4	---
953	Camphene	---	---	0.1	---
978	*β*-Pinene	---	0.1	0.3	---
992	Myrcene	13.9	15.1	tr	---
1004	*α*-Phellandrene	---	---	tr	---
1010	*δ*-3-Carene	---	---	0.3	---
1027	Limonene	---	0.2	---	tr
1031	Benzyl alcohol	0.3	tr	---	tr
1043	Phenylacetaldehyde	---	---	---	tr
1087	Terpinolene	tr	0.2	1.1	---
1097	Linalool	0.5	---	0.2	tr
1184	*p*-Cymen-8-ol	---	---	---	tr
1189	*α*-Terpineol	---	---	---	tr
1374	*α*-Copaene	tr	---	tr	tr
1383	*β*-Bourbonene	0.1	---	0.8	0.6
1391	*β*-Elemene	0.7	---	5.1	3.9
1419	(*E*)-Caryophyllene	10.7	8.2	15.7	12.5
1428	*β*-Copaene	0.2	---	0.2	0.2
1453	*α*-Humulene	2.7	1.9	4.9	4.7
1458	(*E*)-*β*-Farnesene	---	---	0.8	0.7
1478	*γ*-Muurolene	0.2	tr	0.5	0.4
1482	Germacrene D	37.3	28.6	30.2	28.0
1487	(*E*)-*β*-Ionone	0.2	tr	0.3	0.4
1494	*cis*-*β*-Guaiene	---	---	0.7	1.1
1497	Bicyclogermacrene	0.9	0.4	1.4	1.4
1501	*α*-Muurolene	---	---	0.2	0.3
1505	Germacrene A	---	---	2.0	3.6
1510	(*E*,*E*)-*α*-Farnesene	0.6	0.3	1.1	1.1
1514	*γ*-Cadinene	---	---	0.6	0.9

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RI	Compound	Percent Composition			
		Suburban Huntsville		Monte Sano	Wheeler
		#1	#2	Mountain	NWR
1516	Cubebol	---	---	tr	0.9
1525	*δ*-Cadinene	0.4	0.2	2.0	2.4
1566	(*E*)-Nerolidol	1.9	1.2	5.9	10.4
1575	Germacrene D-4-ol	---	---	---	1.0
1578	Spathulenol	---	---	---	tr
1581	Caryophyllene oxide	---	---	0.3	1.4
1593	Salvial-4(14)-en-1-one	---	---	---	tr
1608	Humulene epoxide II	---	---	---	0.2
1614	1,10-di-*epi*-Cubenol	---	---	---	0.4
1617	Junenol	---	---	---	0.2
1627	1-*epi*-Cubenol	---	---	---	0.4
1635	Caryophylla-4(12),8(13)-dien-5-ol	---	---	---	tr
1641	*τ*-Muurolol	---	---	2.0	5.2
1645	*α*-Muurolol (= Torreyol)	---	---	0.3	1.0
1654	*α*-Cadinol	---	---	2.5	5.5
1659	Unidentified oxygenated sesquiterpenoid	---	---	0.2	0.7
1664	Unidentified oxygenated sesquiterpenoid	---	---	0.4	1.2
1669	14-Hydroxy-9-*epi*-(*Z*)-caryophyllene	---	---	---	0.8
1679	Unidentified oxygenated sesquiterpenoid	---	---	---	0.7
1685	Germacra-4(15),5,10(14)-trien-1*α*-ol	---	---	0.4	1.8
1689	Shyobunol	---	---	0.5	2.3
1739	Mint sulfide	---	---	---	0.1
1760	Cyclocolorenone	---	2.8	---	0.4
1798	14-Hydroxy-*δ*-cadinene	---	---	---	0.1
1953	Palmitic acid	---	---	---	0.2
2900	Nonacosane	---	---	---	1.1
	Total Identified	99.8	100.0	98.6	95.6
	Fatty-acid-derivatives	29.4	40.2	16.5	1.2
	Monoterpene hydrocarbons	13.9	16.2	3.2	0.0
	Oxygenated monoterpenoids	0.5	0.0	0.2	0.0
	Sesquiterpene hydrocarbons	53.7	39.6	66.3	61.8
	Oxygenated sesquiterpenoids	1.9	4.0	12.5	34.7
	Others	0.5	0.0	1.0	0.5

[a]"---" = not detected. [b]"tr" = trace (< 0.05%).

3. Results and Discussion

A total of 60 compounds were identified in the *A*. *spinosa* leaf oils, representing 95.6% - 100% of the compositions. *A*. *spinosa* leaf oils from suburban Huntsville, Alabama, were composed largely of sesquiterpene hydrocarbons (53.7% and 39.6%), dominated by germacrene D (37.3% and 28.6%), (*E*)-caryophyllene (10.7% and 8.2%), and α-humulene (1.9% and 2.7%); fatty-acid derivatives (29.4% and 40.2%), mostly (2*E*)-hexenal; and the monoterpene hydrocarbon myrcene (13.9% and 15.1%). The samples growing wild in natural habitats had much reduced monoterpenes but increased concentrations of sesquiterpenoids. The most abundant leaf oil components in the Monte Sano sample were germacrene D (30.2%), (*E*)-caryophyllene (15.7%), (2*E*)-hexenal (13.8%), (*E*)-nerolidol (5.9%), and α-humulene (4.9%). The Wheeler NWR sample of *A*. *spinosa* has similar concentrations of sesquiterpene hydrocarbons to the Monte Sano sample, but virtually no monoterpenoids or "green leaf" volatiles. It was composed, however, of a diverse array of oxygenated sesquiterpenoids including relatively large concentrations of (*E*)-nerolidol (10.4%), *α*-cadinol (5.5%), *τ*-muurolol (5.2%), and shyobunol (2.3%).

Essential oils from other members of the Araliaceae have been analyzed. Pinenes dominated the essential oils

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of *Acanthopanax trifoliatus* [3], *Schefflera heptaphylla* [4], and *Aralia cachemirica* [5], while *δ*-3-carene was the most abundant component of *Dendropanax capillaris* [6]. The sesquiterpene hydrocarbons (*E*)-caryophyllene, *α*-humulene, and germacrene D were the dominant components of *Schefflera stellata* [7], *Schefflera rodrigueziana* [6], and *Oreopanax nubigenus* [6]; (*E*)-nerolidol was abundant in both *Pseudopanax discolor* [8] and *Oplopanax horridus* [9]; and (2*E*)-hexenal was a major component of *Dendropanax gonatopodus* [10]. The essential oils of *Pseudopanax discolor* and *Pseudopanax lessonii* had notable quantities of *τ*-muurolol [8], while *Dendropanax arboreus* and *Oreopanax xalapensis* were rich in shyobunol [11].

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