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Volatile profile of *Calycanthus occidentalis* achenes and evidence for a diverse range of semiochemicals for vespicochory by pestiferous *Vespula pensylvanica*

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ABSTRACT

Vespidae represents a large and diverse family and comprises both social and solitary wasps that occupy a large array of habitats. Though some are considered nuisance insects, many of the species are also known as insect predators and pollinators. An additional role of select vespid wasps, originally elucidated approximately 30 years ago and gaining in the number of identified wasps, is dispersal of plant reproductive organs (vespicochory). This relationship is considered mutualistic given the long-range dispersal benefit to the diaspore, and the nutritional benefit to the wasps. Long-chain hydrocarbons have been shown to be responsible for attracting wasps to the organs of some diaspores. Recent observations in northern California indicated that yellowjackets (*Vespula* spp.), particularly western yellowjacket, *Vespula pensylvanica*, procured and transported diaspores (achenes) from the western spicebush (*Calycanthus occidentalis*), a riparian shrub endemic to California. Volatile profile analyses of *C. occidentalis* achenes showed emission of a diverse range of volatile classes. Empirical electrophysiological and trapping studies suggest a composite of odors may stimulate wasp attractancy to the achene.

KEYWORDS: attractant, diaspore, achene dispersal, semiochemicals, yellowjacket, vespicochory.

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INTRODUCTION

Yellowjackets of the genus *Vespula* are a widespread group of eusocial wasps found throughout the northern hemisphere. The western yellowjacket, *Vespula pensylvanica* (de Saussure) is a ground-nesting pestiferous social wasp native to the western U.S. [1]. In 1979 *V. pensylvanica* was documented in Hawaii and Maui [2], and it was quickly realized that this invasive species required control measures [3, 4] to alleviate pestiferous behavior toward humans, as well as ecological damage. In contrast to these invasive and bothersome characteristics, *V. pensylvanica* provides ecological benefits such as arthropod predation [5] and floral pollination [4].

A more recently identified ecological role of *V. pensylvanica* behavior has been vespicochory – the ability to transport and hence disperse plant reproductive organs. Reproductive organs dispersed in this way have an elaiosome containing proteins, fat, carbohydrates, and simple sugars [6, 7]. Plant reproductive organ dispersal is performed by a number of abiotic (e.g., wind, water) and biotic (e.g., ants, birds, rodents) means; however, dispersal by vespids is a relatively newly discovered phenomenon, with vespicochory reported for *Vespula vulgaris* [8, 9], *Vespula maculifrons* [10], *Vespa affinis* [11], and *Vespa velutina* [12-14]. *Vespula pensylvanica* were recently observed dispersing achenes of the California- endemic shrub *Calycanthus occidentalis* in northern California [7]. Table 1 summarizes documented observed instances of plant reproductive organ dispersal by Vespidae.

Table 1. Reported cases of vespicochory, including species of plants and wasps involved.

Vespidae (genus sp.)	Plant	State/Country	Ref.
Vespula vulgaris	*Vancouveria hexandra*	Washington, USA	[8]
Vespula vulgaris	*Trillium ovatum*	Oregon, USA	[9]
Vespula maculifrons	*Trillium cuneatum* *T. undulatum* *T. catesbaei*	North Carolina and South Carolina, USA	[10]
Vespa affinis	*Aquilaria malaccensis*	India (north east)	[11]
Vespa velutina	*Aquilaria sinensis* *Stemona tuberosa*	China China	[12] [14]
Vespula pensylvanica	*Calycanthus occidentalis*	California, USA	[7]

One possible mechanism for vespid attraction to these plant organs has recently been described in an elegant study by Chen and co-workers [14]. In their work, they describe a mixture of long-chain hydrocarbons from *Stemona tuberosa* elaiosomes as the odors responsible for attracting *Vespa velutina*. Other semiochemicals of vespids are well documented and include commercially available traps (e.g., Rescue!®) containing heptyl butyrate [15, 16], kairomones from insect-produced honeydew [17], fungal-produced volatiles [18], odors from cooked meats [19], and combining heptyl butyrate with chicken extract [20], among others. Long-chain cuticular hydrocarbons similar to those extracted from elaiosomes [14] have also been extracted from the bodies of species of yellowjackets, including *V. pensylvanica* [21].

Calycanthus occidentalis Hook. and Arn. (Laurales: Calycanthaceae), commonly known as the spicebush, is a shrub found in the foothills of the Coast, Cascade, and Sierra Nevada mountain ranges in northern California [22]. Spicebush is dependent upon insects for pollination [22], yet no literature exists regarding dispersal of its reproductive organs (achenes). The leaves and twigs of spicebush have been steam distilled and the chemical composition reported [23] to contain primarily cineol, linalyl acetate, borneol, and pinene, among trace amounts of camphor, methyl salicylate, and other sesquiterpene alcohols. No literature currently exists for *C. occidentalis* achene odors. The objectives of this research were to 1) obtain the volatile profile of *C. occidentalis* achenes, 2) survey germane classes of compounds for their ability to elicit electrophysiological responses from *V. pensylvanica* antennae, and 3) probe wasp attractancy via field trapping studies of select chemical classes derived from the seed achene profile and electrophysiological studies.

MATERIALS AND METHODS

Achene collections

Achenes used for volatile collections were from the same batches as those collected for wasp observational and behavioural trials as described in Burge and Beck [7], with vouchers deposited at the University California, Davis Center for Plant Diversity. Collected achenes were transported overnight from California to the USDA-ARS Florida laboratory in sealed brown paper bags or sealed 4 or 11-mL scintillation vials with modified lids as described in Beck *et al*. [24].

Wasp collections

Individual *V. pensylvanica* were captured at locations in northern California [7] and placed in small plastic containers with modified lids for air exchange. In each container a cotton ball soaked in sugar water was used to maintain the wasps during shipment. The containers were placed in insulated boxes and shipped overnight to the USDA-ARS Florida laboratory for electrophysiological studies. Voucher specimens (D.O. Burge 1024) were deposited at the University of California, Davis, Bohart Museum of Entomology, as well as with the Florida Department of Agriculture and Consumer Services, Division of Plant Industry, Gainesville, Florida.

Achene volatile collection and analysis

Calycanthus occidentalis achenes (ca. 100) were placed in Wheaton 11-mL vials with a modified cap containing a septum and Teflon liner, similar

to published methods [24]. The vials were capped and placed in an oven at 40 °C to mimic warm weather conditions and facilitate full volatile emission from the achenes. Volatiles were allowed to collect (permeate) in the closed system for 60 min at 40 °C before insertion of a solid-phase microextraction (SPME, PDMS-DVB, Supelco, Bellefonte, PA, USA) fiber, which was exposed to the headspace volatiles for 30 min at 40 °C, removed, and immediately thermally desorbed onto an Agilent 7890A gas chromatograph (GC) coupled to a 5975C MSD detector in electron ionization mode (Palo, Alto, CA, USA) outfitted with a J&W Scientific (Folsom, CA, USA) DB-Wax column (60 m x 320 μm x 0.25 μm) [25]. Each achene collection from the three sites (Table 2) was analysed in triplicate, using a separate set of 100 achenes. For identification and authentication purposes, SPME volatile collections were additionally analysed on an Agilent 7890B GC coupled to a 5977B MSD detector in electron ionization mode, and outfitted with a J&W Scientific DB-1 column (60 m x 320 μm x 0.25 μm). Retention index (RI) values were calculated using a homologous series of *n*-alkanes on both the DB-1 and the DB-Wax columns. RI values from both columns were used to assist with initial identification, and identities were further confirmed by comparison to retention times and fragmentation patterns of standards. Compound identities not verified on both instruments with a commercial or other available standard were marked as tentatively identified, and not included in Table 2. For volatile profile and bioassay result comparison, *C. occidentalis* achenes were also chemically extracted using the method identical to Burge and Beck [7]. Volatile profiles for the SPME and chemical extraction methods were nearly identical, aside from the expected heavy alkanes extracted from the chemical extraction. Peaks identified as background from the containers, fibers, and columns detected in blanks were removed before analysis.

Electrophysiological studies

Using methods similar to previously published protocols for honeybees [25], both *V. pensylvanica* antennae were excised and placed on the fork holder using electrode gel. Bioassay discs were loaded with 50 μg of each test component using 10 μL of solutions of 5 μg/μL in pentane. After solution addition, the pentane was allowed to evaporate for 1 min and the discs placed in a Pasteur pipette, which was then inserted into a volatile tube leading to the insect antennae. All electrophysiological studies were performed using a 4-channel acquisition controller, electrode fork holder, and pre-amplifier (Syntech, Kirchzarten, Germany). A 0.5 s pulse flow (300 mL/min) and a humidified continuous flow (125 mL/min) directed odors through an air and volatile tube (1.5 cm diameter) containing the mounted antennae and probe. A Faraday cage was used to protect against ambient electrical interference. To account for variability in response among individuals, responses to blanks (10 μL pentane loaded onto a bioassay disc) were subtracted from each sample and antennal response values then normalized to the standard stimulus (50 μg of benzaldehyde) set at 100% (Table 3).

Field trapping studies

Wasp capture used identical techniques as previously published [7] using Rescue!® traps, but instead of the achene extracts loaded onto bioassay discs as in Burge and Beck [7], pure individual compounds or blends of pure compounds were loaded into 2.0 mL conical vials and sealed until the field trials. Prior to placement in the field, the vial caps were replaced with caps containing a 2 mm hole, and with cotton strips (ca. 1 x 3 cm) inserted. For all trapping studies, each compound was evaluated with no solvents to dilute the compounds or the blends (neat). For individual compounds, 180 μL were added into each vial (n = 5 or 6) for each lure (Table 4). For blends, the total volume added was 180 μL; thus, a three-component mixture comprised 60 μL of each compound. Traps baited with heptyl butyrate were used as a positive control to confirm wasp population at each site [7]. Field trapping trials were performed at the University of California La Kretz Center Field Station, located in the western Santa Monica Mountains (Trial 1; Table 4; 34.0970, -118.8156) and at Elysian Park, located in the eastern Santa Monica Mountains (Trial 2; Table 4; 34.0841, -118.2462).

RESULTS AND DISCUSSION

The volatile profiles of the achenes were surprisingly high in the number of compounds and chemical class diversity. A total of 102 compounds were detected consistently in the three batches of achenes analysed by SPME GC-MS. Table 2 provides the 44 authenticated compounds reported by relative abundances, and illustrates the wide diversity

Table 2. Authenticated volatiles produced by *C. occidentalis* achenes. Volatile peak areas reported are averages (n = 3) from each of the three locations [7] and are relative abundances from GC-MS analysis. Volatiles are reported in descending order of average detected amounts.

Compound	RI[a]		Peak Area (± sem)					
	DB-wax	DB-1	Kaweah River (DB-2201)		Butte Creek (DB 2122)		Hooker Creek (DB 2123)	
nonanal[b]	1394	1083	1,289,401,087	(244,552,234)	727,344,459	(67,137,093)	487,051,146	(42,928,330)
2,5-dimethyl-pyrazine	1322	885	558,787,391	(46,812,239)	487,622,536	(55,874,018)	732,462,146	(33,695,464)
3-ethyl-2,5-dimethyl-pyrazine[b]	1447	1057	468,699,797	(27,713,972)	364,465,166	(46,454,642)	662,852,152	(31,798,268)
2,6-dimethyl-pyrazine	1329	885	131,681,985	(24,707,451)	110,345,785	(19,771,620)	298,042,423	(28,829,615)
hexanal	1080	776	135,435,398	(14,933,511)	248,082,702	(84,315,646)	111,902,744	(9,341,147)
benzaldehyde	1520	929	82,977,648	(8,450,247)	101,018,513	(3,480,147)	91,242,111	(12,540,419)
cineol	1209	1021	122,764,947	(80,890,831)	37,689,026	(18,214,316)	41,250,000	(7,760,745)
2-nonanone	1389	1072	19,956,920	(7,183,954)	89,117,400	(24,511,228)	58,794,443	(7,036,621)
octanal	1289	982	51,187,311	(6,900,192)	50,900,619	(9,975,779)	34,399,795	(3,057,536)
2-heptanone	1181	869	13,904,082	(2,359,512)	54,121,556	(11,815,415)	41,010,689	(10,143,855)
benzyl alcohol	1876	1003	28,703,178	(3,959,248)	27,509,816	(3,284,716)	28,740,829	(3,507,662)
2-methyl-butanal	913	641	20,104,206	(3,203,941)	32,007,494	(4,399,082)	31,858,374	(4,303,128)
phenylacetaldehyde	1640	1007	23,182,816	(2,215,610)	30,114,097	(5,844,100)	30,637,427	(3,232,366)
acetophenone	1648	1034	27,559,493	(3,560,375)	28,239,101	(3,995,242)	27,885,013	(4,065,726)
2-ethyl-1-hexanol[c]	1493	1014	12,916,645	(3,241,442)	20,568,800	(2,759,299)	41,004,423	(4,558,562)
decanal	1499	1185	28,448,055	(2,368,074)	26,649,762	(2,228,380)	16,580,870	(918,683)
2-methyl-1-butanol	1208	722	8,787,104	(3,043,265)	28,960,758	(8,660,369)	33,250,000	(1,701,715)
phenylacetonitrile	1923	1089	25,926,268	(2,240,781)	31,440,920	(4,145,977)	8,332,060	(2,787,440)
2*E*-hexenal[b]	1217	826	13,633,539	(1,763,784)	25,189,946	(8,795,089)	18,381,867	(1,442,477)
bornyl acetate	1581	1271	7,679,169	(3,378,794)	18,274,993	(1,945,235)	29,627,975	(7,294,840)
linalool	1550	1085	21,956,951	(3,963,022)	6,342,216	(939,212)	23,940,714	(5,471,560)
D-limonene	1199	1023	14,396,440	(2,722,757)	22,573,803	(7,733,596)	12,865,055	(1,793,386)
2-methyl-1-propanol	1092	611	7,082,302	(1,376,103)	17,892,115	(3,429,567)	21,235,212	(1,749,264)
α-pinene	1021	933	14,379,912	(2,936,381)	17,343,905	(6,549,905)	10,551,700	(2,004,597)
2,3-butanediol	1578	765	10,749,220	(1,080,925)	11,607,293	(1,990,831)	16,933,961	(1,667,412)
6-methyl-5-hepten-2-one	1337		12,804,742	(1,992,263)	12,683,554	(2,079,966)	12,700,341	(2,205,843)
1-methyl-2-pyrrolidinone	1675		11,604,379	(1,894,298)	14,531,438	(2,994,121)	9,711,788	(1,170,547)

Table 2 continued..

2*E*-octenal	1429	1033	9,390,498	(1,707,095)	17,711,960	(8,323,603)	6,937,043	(698,616)
β-pinene	1106	972	13,428,313	(3,807,782)	14,658,589	(4,497,170)	5,946,845	(973,565)
2-undecanone	1599	1274	8,695,027	(1,017,617)	13,457,598	(2,463,626)	11,018,321	(3,712,610)
phenylethyl alcohol	1911		7,091,904	(1,338,105)	7,347,616	(680,330)	11,775,461	(1,886,256)
1-hexanol	1356	854	4,211,231	(559,731)	10,588,097	(1,611,894)	7,060,672	(671,726)
propylene glycol	1591		5,759,004	(1,074,734)	9,542,752	(1,072,049)	5,577,428	(1,060,460)
1-pentanol	1252	750	3,612,930	(236,564)	7,349,320	(310,622)	6,133,227	(845,226)
decane	999		4,931,943	(1,011,488)	4,813,863	(2,418,401)	4,553,671	(1,299,664)
methyl salicylate	1773	1169	4,324,596	(836,769)	3,958,639	(233,231)	3,755,929	(1,453,084)
octane	800		3,018,164	(369,613)	3,250,790	(1,221,903)	4,308,764	(1,221,385)
camphene	1062	946	2,996,881	(1,828,300)	4,804,136	(3,922,317)	2,727,455	(1,054,043)
2-butanone	902		2,994,953	(947,197)	3,914,873	(555,478)	3,603,644	(515,923)
tetramethyl pyrazine	1477		2,368,857	(711,873)	2,303,020	(1,379,745)	5,373,996	(373,566)
acetoin	1283		1,767,812	(594,671)	2,335,906	(239,102)	2,953,818	(73,413)
2-octanone	1285	971	1,021,181	(441,950)	3,018,199	(560,658)	2,839,804	(276,349)
ethanol	932		919,144	(346,284)	1,648,175	(859,646)	3,824,424	(387,546)
methyl butyrate	824		880,109	(78,077)	856,315	(102,422)	1,292,519	(246,463)

[a]Retention indices were calculated using a homologous series of *n*-alkanes.
[b]Compound used in electroantennographic (EAG) and behavioral studies.
[c]In DB-Wax, compound co-eluted with methyl nonanoate.

Table 3. Electrophysiological studies of representative classes of detected compounds from *C. occidentalis* achenes or purported semiochemicals, and elicited responses from *V. pensylvanica* antennae.

Compound class	Compound	Antennal response (%, *n* = 6-8 wasps)[a]
long-chain alkane	tetracosane	22.6 ± 9.1
	nonacosane	24.2 ± 11.8
aldehyde	(2*E*)-hexenal[b]	290.3 ± 63.2
	nonanal[b]	146.9 ± 55.8
acid	octanoic acid[b]	150.0 ± 65.7
alcohol	1-octanol[b]	273.0 ± 65.2
aromatic	2-ethyl-3,5-dimethylpyrazine[b]	613.8 ± 537.1

[a]Normalized electroantennographic (EAG) response: sample – blank, and percentage of positive control, benzaldehyde, average ± sem
[b]Either that compound or the class of compounds was detected

Table 4. Field trapping studies of *V. pensylvanica* for select classes detected from *C. occidentalis* achenes, or purported semiochemicals (*n* = 5, trial 1; *n* = 6, trial 2).

Compound or blend	Trial 1	Trial 2	Combined
tetracosane (24)	1.0 ± 0.5	6.8 ± 3.2	4.2 ± 1.9
nonanal (9AL)	1.6 ± 0.5	9.5 ± 7.1	5.9 ± 3.9
1-octanol (8OH)	5.2 ± 1.7	16.8 ± 9.4	11.6 ± 5.3
2-ethyl-3,5-dimethylpyrazine (EDMP)	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
blend 24/9AL/8OH/EDMP	-	0.3 ± 0.2	0.3 ± 0.2
blend 24/9AL/8OH	-	1.2 ± 0.2	1.2 ± 0.5

of chemical classes detected. Generally classified but not fully authenticated, the total profile comprised: 17 aromatics (e.g., furans, pyrazines); 16 aldehydes; 13 ketones; 11 alkenes; 7 benzenoids; 7 monoterpenes; 7 primary alcohols; 5 various alkanes; 2 each of sesquiterpenes and unknown compounds; and, 1 each of other classes.

Given the unexpectedly large number of detected compounds and range of classes, it is surprising that the western yellowjacket (*V. pensylvanica*) is known to be attracted to a single compound, namely heptyl butyrate (a simple ester) [15, 16]. This is nicely demonstrated by the success of the commercial attractant Rescue!® which contains only heptyl butyrate. However, when presented with a complex bouquet of volatiles emitted from a food source, there is likely more than one compound or class of compounds that provides the attractiveness of the food source. Indeed, *V. pensylvanica* were shown to be attracted to the primary alcohols 2-methyl-1-butanol, 3-methyl-1-butanol, and 2-phenylethanol, volatiles produced by fungal colonies of *Aureobasidium pullulans* [18]. Moreover, a relatively complex mixture of volatiles from the honeydew of insects was shown to attract *V. vulgaris* [17]. The authors of this study [17] also demonstrated wasp attraction to simpler blends including aromatics, an aldehyde, a secondary alcohol, and a primary alcohol. Conversely, Zhang and co-workers [26] showed repellency of *V. pensylvanica* using essential oils that included compound classes of terpenoids, aromatics, and a secondary alcohol. Interestingly, in their 2013 repellency study [26] methyl salicylate and 3-octanol were noted as providing significant repellency of *V. pensylvanica*, whereas Brown's 2015 study [17] on *V. vulgaris* noted these two compounds as

slight attractants when presented individually during trapping studies. Semiochemical activity (either attractant or repellent) of the primary alcohol 1-octanol was corroborated in the present study (Table 3) and also attracted the most *V. pensylvanica* in our trapping studies (Table 4).

This dichotomy of class and compound semiochemical behavior was also indirectly noted in the pyrazine class of compounds, which as reported in Table 2 were amongst the most highly emitted compounds from *C. occidentalis* achenes. Literature regarding *V. pensylvanica* attraction to cooked meat is abundant, and cooked meat is known to be a source of proteins [3, 19, 20]. Interestingly, it is also known in the literature that pyrazines (and aldehydes) are commonly found in the headspace of cooked meats [27, 28]. In the present study, the compound 2-ethyl-3,5-dimethylpyrazine elicited the highest response from *V. pensylvanica* antennae (Table 3), yet when tested individually during wasp trapping studies (Table 4) this compound did not attract any *V. pensylvanica*. When included in a blend of volatiles with other tested compounds, the results suggested that 2-ethyl-3,5-dimethylpyrazine may inhibit attraction of *V. pensylvanica*.

While little is known about the role of pheromones in vespids [29], pyrazines have been reported as sex pheromones of Eurytomidae [30] and Thynnidae wasps [31]. However, long-chain hydrocarbons have been detected on the cuticles of several species of yellowjackets, including *V. pensylvanica* [21], and long-chain hydrocarbons have also been shown to signal fertility in some vespids [32]. Interestingly, several long-chain hydrocarbons were detected in the elaiosomes of *Stemona tuberosa*, a seed dispersed by *Vespa velutina* [14]. Further work by the same authors showed that these same long-chain hydrocarbons were the volatiles involved in the attraction of Vespa [14]. In the present study we confirmed both the electrophysiological activity (Table 3) and attractancy (Table 4) of the long-chain hydrocarbon tetracosane, a component detected in the Chen *et al.* [14], the Derstine *et al.* [21], and van Zweden *et al.* [32] studies.

CONCLUSION

The broad range of volatile compound classes detected from the achenes of *Calycanthus occidentalis*, and the noted electrophysiological and behavioral responses provide evidence for a potential complex blend of semiochemicals for *C. occidentalis* vespicochory. This hypothesis is supported by the association of some of these food-related compounds detected in the present study to known attractants of *V. pensylvanica*. Some examples include: 1) pyrazines, which are indicative of a source of protein, albeit the trapping study results for the chosen pyrazine suggested an inhibitory activity, 2) the aldehydes hexanal, nonanal, and octanal, which are indicative of lipid oxidation [28], and 3) the primary alcohols phenylethanol and 2-methyl-1-butanol, which have been detected from a fungus that has been reported as a source of food for wasps [18]. Further investigations into the volatile profiles of the plant reproductive organs from the first four studies [8-11] listed in Table 1, along with in-depth electrophysiological and behavioral studies may reveal definitively the identities of the semiochemicals responsible for their noted vespicochory.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

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