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Short Communication

Composition of essential oil from naturally exuded *Pinus ponderosa* var. *ponderosa* (Pinaceae) resin

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Abstract

Pinus ponderosa is an essential oil-bearing tree of the Pinaceae family. Naturally exuded resin was collected from *P. ponderosa* trees, hydrodistilled to isolate the volatile fraction (essential oil), and analyzed by GC/MS to establish its chemical profile. The resulting essential oil samples (n = 10) had an average yield (w/w) of 6.14% and were largely composed (average values) of δ-3-carene (44.7%), β-pinene (20.9%), α-pinene (7.5%), limonene (4.6%), myrcene (4.2%), and 3,7,7-trimethyl-1,3,5-cycloheptatriene (2.7%). Tree resins are an important raw material source for a short list of essential oils. However, to the best of our knowledge, this is the first study to investigate the hydrodistilled *P. ponderosa* naturally exuded resin, providing a foundation for future research on the production and application of a novel essential oil product.

1. Introduction

Pinus ponderosa Douglas ex C. Lawson (ponderosa pine) is an essential oil-bearing tree in the family Pinaceae [1]. Ponderosa pine is native to the mountainous regions of western North America, and the species is further distinguished into three varieties (var. *ponderosa*, var. *arizonica*, var. *scopulorum*) [2]. Within the western mountainous region of the United States, specifically in west-central Idaho, the native variety of ponderosa pine is *P. ponderosa* var. *ponderosa* [3-5] (Fig. 1).

Historically, ponderosa pine has been used as a lumber and fuel source [6] and pine resin essential oils were used as a solvent in various industrial processes [7]. Pine resin is composed of a non-volatile fraction (resinous acids, etc.) and a volatile fraction (essential oil) [7].

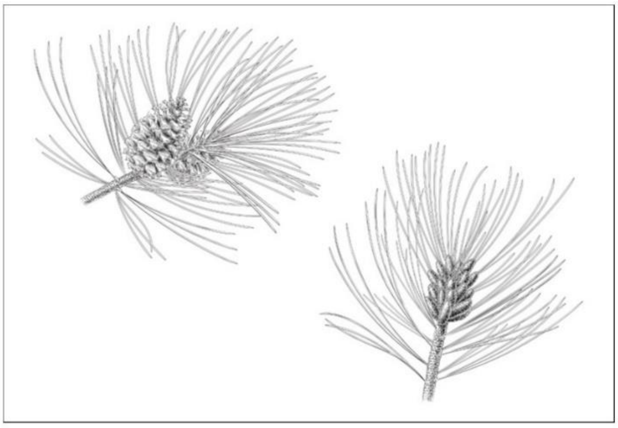

Figure 1. Illustration depicting *Pinus ponderosa* var. *ponderosa* needles with female cones (left) and male cones (right), which are found on the same tree. Botanical illustration by Rick Simonson (Science Lab Studios, Inc.).

Pinus ponderosa needle (leaf) essential oil has been shown to be largely composed of α-pinene, β-pinene, δ-3-carene, α-terpineol, and methyl chavicol [8, 9].

Table 1. Hydrodistillation and essential oil production details, including resin mass (g), essential oil yield (g), and essential oil % (w/w).

Products	*Pinus ponderosa* sample									
	A	B	C	D	E	F	G	H	I	J
Resin mass (g)	371.32	379.34	187.03	275.21	224.83	112.74	173.77	331.32	284.96	78.48
Essential oil yield (g)	23.80	31.00	5.81	16.81	15.10	7.20	3.31	19.58	22.57	6.91
Essential oil % (w/w)	6.41	8.17	3.11	6.11	6.72	6.39	1.90	5.91	7.92	8.80

P. ponderosa resin essential oil has been shown to be largely composed of three of the same compounds (α-pinene, β-pinene, δ-3-carene), and, additionally, myrcene and limonene [10, 11]. To the best of our knowledge, this is the first comprehensive investigation of hydrodistilled *P. ponderosa* var. *ponderosa* resin essential oil, specifically of the naturally exuded resin.

2. Materials and methods

Pinus ponderosa resin was collected from 10 individual trees on 17 September 2024 from native populations. The collection site is located on private lands in Idaho County, Idaho (USA) and borders the Salmon River (560 m elevation); however, respecting the landowner's request, specific GPS coordinates were withheld. A representative voucher sample was produced from the said site and is held in the Young Living Aromatic Herbarium (YLAH): *P. ponderosa* var. *ponderosa*, Wilson 2024-01.

Essential oil (EO) samples (n = 10) were produced by distilling the resin from 10 individual trees. Samples were produced by laboratory-scale hydrodistillation as follows: 6 L of water was added to the bottom of a 12-L distillation chamber (Albrigi Luigi S.R.L., Grezzana, Italy), resin was accurately weighed and added to the distillation chamber, hydrodistilled for 3 h, and volatile oil was separated with a cooled condenser and Florentine flask. The EO samples were filtered and stored in sealed amber glass bottles at room temperature until analysis.

To determine volatile compound profiles, EO samples were analyzed, and compounds were identified and quantified by GC/MS using an Agilent 7890B GC/5977B MSD (Agilent Technologies, Santa Clara, CA, USA) and Agilent J & W DB-5, 60 m × 0.25 mm, 0.25 µm film thickness, fused silica capillary column. Operating conditions: 0.1 µL of sample (20% soln. for EO in ethanol), 100:1 split ratio, initial oven temp. of 40 °C with an initial hold time of 5 min, and oven ramp rate of 4.5 °C per min to 310 °C with a hold time of 5 min. The electron ionization energy was 70 eV, the scan range was 35–650 amu, the scan rate was 2.4 scans per s, source temp. 230 °C, and quadrupole temp. 150 °C. The compounds were identified using the Adams volatile oil library [12] and a Chemstation library search in conjunction with retention indices (KI). Note that limonene/β-phellandrene and terpinen-4-ol/m-cymen-8-ol elutes as single peaks. Their amounts were determined by the ratio of m/z 77/93 (β-phellandrene), 68/79 (limonene), 93/111 (terpinen-4-ol), 43/135 (m-cymen-8-ol).

The percent yield was calculated as the ratio of the mass of essential oil produced to the mass of processed plant material immediately before hydrodistillation, multiplied by 100.

3. Results

Hydrodistillation of resin from 10 individual trees of *Pinus ponderosa* resulted in 10 essential oil (EO) samples, identified as samples A-J. Naturally exuded resin was collected from the trunk and branches of each tree, up to 2 m above the ground. The yields (w/w) ranged from 1.90-8.80%, with an average of 6.14% (Table 1).

The GC/MS analysis identified 49 volatile compounds that were present in at least one sample (Table 2). Prominent compounds, defined as being ≥ 2% (relative area%) in at least one sample, included (average values) δ-3-carene (44.7%), β-pinene (20.9%), α-pinene (7.5%), limonene (4.6%), myrcene (4.2%), and 3,7,7-trimethyl-1,3,5-cycloheptatriene (2.7%). These same compounds displayed high variability in their relative abundance between samples (α-pinene (σ = 4.4), 3,7,7-trimethyl-1,3,5-cycloheptatriene (σ = 2.6), β-pinene (σ = 10.7), myrcene (σ = 2.4), δ-3-carene (σ = 12.9), and limonene (σ = 3.9)).

Table 2. Essential oils profiles of *Pinus ponderosa* resin samples.

KI	Compounds	*Pinus ponderosa* resin essential oil (area%)									
		A	B	C	D	E	F	G	H	I	J
921	Tricyclene	nd	tr	tr	tr	tr	tr	tr	tr	nd	tr
924	α-Thujene	0.3	0.4	0.4	0.2	0.3	0.3	0.5	0.7	0.6	0.1
932	α-Pinene	2.3	14.0	9.9	11.0	14.9	6.1	4.9	3.2	2.8	6.3
946	Camphene	0.1	0.2	0.3	0.3	0.3	0.2	0.1	0.1	0.1	0.2
953	2,4,(10)-Thujadiene	tr	tr	tr	tr	tr	tr	tr	nd	nd	nd
966*	3,7,7-Trimethyl-1,3,5-Cycloheptatriene	9.0	1.5	6.2	1.7	1.5	1.2	1.0	3.2	1.3	0.2
969	Sabinene	0.3	0.3	0.2	0.2	0.3	0.3	0.4	0.2	0.4	0.4
974	β-Pinene	7.8	28.8	25.9	32.6	26.4	30.8	18.2	4.6	4.4	29.9
988	Myrcene	0.6	2.9	0.7	2.9	3.6	4.1	7.4	5.6	6.3	7.4
1002	α-Phellandrene	tr	tr	0.1	0.1	0.1	0.1	tr	0.1	0.1	tr
1008	δ-3-Carene	45.8	35.1	38.9	34.4	36.6	44.1	51.2	64.7	69.5	27.0
1014	α-Terpinene	0.1	0.1	0.1	0.1	tr	0.1	0.1	0.2	0.2	0.1
1020	p-Cymene	0.9	0.1	0.4	0.1	0.2	0.1	0.1	0.2	0.1	tr
1022	o-Cymene	3.6	0.5	1.7	0.6	0.7	0.8	0.8	1.0	0.6	0.2
1024	Limonene	3.0	9.4	5.6	10.9	5.0	0.9	0.8	0.6	0.5	9.4
1025	β-Phellandrene	nd	nd	nd	nd	nd	0.6	0.6	0.4	0.4	0.6
1054	γ-Terpinene	0.3	0.2	0.1	0.1	0.2	0.3	0.4	0.4	0.4	0.2
1085	p-Mentha-2,4(8)-diene	nd	0.1	nd	0.1	0.1	0.1	0.2	0.2	0.2	0.1
1086	Terpinolene	1.0	1.4	0.7	1.2	1.3	1.8	2.8	2.6	3.1	1.7
1095	Linalool	4.2	0.1	nd	0.1	nd	0.3	0.1	tr	nd	tr
1100	n-Undecane	nd	nd	0.2	nd	nd	nd	nd	nd	nd	nd
1114	Endo-Fenchol	0.1	tr	0.1	tr	tr	nd	nd	nd	nd	nd
1122	α-Campholenal	nd	nd	tr	nd	nd	nd	nd	nd	nd	nd
1135	(E)-Pinocarveol	0.2	0.2	0.4	0.2	0.3	0.2	0.1	nd	nd	0.1
1166	p-Mentha-1,5-dien-8-ol	0.1	0.1	0.1	tr	tr	0.2	0.1	0.1	tr	nd
1174	Terpinen-4-Ol	0.8	0.1	0.2	0.1	tr	0.1	0.2	0.2	0.1	tr
1176	m-Cymen-8-ol	1.2	0.1	0.2	0.1	0.1	0.1	0.1	0.1	nd	nd
1179	p-Cymen-8-ol	0.6	0.1	0.2	0.1	0.1	0.1	0.1	0.1	tr	nd
1186	α-Terpineol	1.1	0.1	0.7	0.4	0.1	0.2	0.3	1.7	0.1	0.1
1195	Methyl chavicol	2.2	1.1	1.5	1.0	1.1	1.3	1.5	1.8	1.6	1.1
1346	α-Terpinyl acetate	0.6	0.2	0.2	0.2	0.1	0.3	0.4	0.2	0.1	nd
1348	α-Cubebene	0.2	0.1	tr	nd	0.1	tr	0.1	0.2	0.1	0.1
1350	α-Longipinene	0.3	tr	0.1	tr	tr	0.2	0.1	0.1	0.1	0.1
1373	α-Ylangene	0.2	tr	nd	nd	0.1	tr	0.1	0.1	tr	0.1
1374	α-Copaene	0.6	0.1	nd	nd	0.2	nd	0.2	0.3	0.2	0.2
1387	β-Bourbonene	0.1	0.5	0.2	tr	1.6	0.6	1.0	0.8	1.2	1.5
1403	Methyl eugenyl	0.2	nd	nd	tr	nd	nd	nd	nd	nd	nd
1407	Longifolene	4.2	0.4	1.6	0.4	0.2	2.1	1.6	0.9	0.8	0.8
1419	β-Ylangene	nd	0.1	tr	nd	0.2	nd	0.2	0.2	0.1	0.3
1478	γ-Muurolene	1.3	0.2	0.1	nd	0.7	0.3	0.7	1.0	0.6	1.1
1480	Germacrene D	nd	0.2	nd	nd	0.3	0.3	0.5	0.5	1.0	6.6
1500	α-Muurolene	0.3	0.1	tr	nd	0.5	0.1	0.2	0.6	0.6	0.5
1513	γ-Cadinene	0.4	0.1	0.1	nd	0.2	0.1	0.3	0.4	0.2	0.5
1522	δ-Cadinene	0.9	0.2	0.2	nd	0.7	0.3	0.8	1.0	0.8	1.4
1537	α-Cadinene	nd	nd	nd	nd	tr	nd	tr	tr	tr	0.1
1544	α-Calacorene	0.1	nd	nd	nd	tr	nd	tr	tr	nd	nd
1594	Salvia-4(14)-en-1-one	0.1	nd	nd	nd	tr	nd	tr	nd	nd	tr

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Table 2. *(continued).*

KI	Compounds	*Pinus ponderosa* resin essential oil (area%)									
		A	B	C	D	E	F	G	H	I	J
1968	Sandaracopimara-8(14),15-diene	0.3	tr	0.1	nd	tr	tr	tr	nd	nd	0.2
2184	Sandaracopimarinal	nd	nd	tr	nd	nd	nd	tr	nd	nd	0.1
Identified Total		***95.3***	***98.9***	***97.8***	***99.4***	***98.2***	***98.6***	***98.2***	***98.0***	***98.8***	***98.7***

(*n* = 10). KI is the Kovat's Index value and was previously calculated by Robert Adams using a linear calculation on a DB-5 column [12]. *KI not included in the Adam's Library [12] and was manually calculated using alkane standards on a DB-5 column. The compound name and value (relative area %) are reported for each detected compound. Values less than 0.1% are denoted as trace (tr) and those not detected as nd.

4. Discussion

Collected resin demonstrated an assortment of organoleptic characteristics, ranging from lightly colored, fresh, and semi-liquid to darkly colored, aged, and hard. The variation in the essential oil yield is likely due to the variability in the starting materials, particularly the age of the resin. Future research should focus on the volatile profile, and abundance of volatile compounds, in fresh versus aged resins.

GC/MS analysis resulted in the identification of 49 compounds present in the samples. However, the six prominent compounds previously mentioned (α-pinene, 3,7,7-trimethyl-1,3,5-cycloheptatriene, β-pinene, myrcene, δ-3-carene and limonene) comprised, on average, 84.6% of the volatile profile of each sample. Additionally, as mentioned previously, these same six compounds displayed high variability in their relative abundance between samples. This may also be related to the age of the resin and should be the focus of future research. An interesting relationship was observed between pinenes (sum of α- and β-pinene) and δ-3-carene, in that their sum was on average 73.2% of the total relative area % of each sample and their ratio (sum of pinenes to δ-3-carene) displayed a somewhat inverse relationship (Fig. 2).

Conifer resins within the family Pinaceae (different genera) have also been shown to be largely composed of α-pinene, β-pinene, δ-3-carene, and limonene [13-15]. However, each profile is also distinguished by the additional abundant and variable compounds. Research has been previously conducted on the essential oil profile of *Pinus ponderosa*; however, this previous research was conducted on other plant parts [8,16] and/or using different extraction techniques [10,11]. Previous research on the needle essential oil from *Pinus ponderosa* var. *ponderosa* (origin: central Oregon) is primarily composed of β-pinene (37.4%) and methyl chavicol (25.8%) but has comparatively little δ-3-carene (5.1%) [8]. The needle essential oil profile from *P. ponderosa* var. *brachyptera* (origin: north-central Arizona) also differs from that of *P. ponderosa* var. *ponderosa* resin essential oil from the current study. The essential oil of this needle is largely composed of α-pinene (15.2%) and germacrene D (13.6%) [16]. *P. ponderosa* resin essential oil has been shown to be largely composed of α-pinene, β-pinene, δ-3-carene, myrcene, and limonene [10,11]. Differences in these essential oil profiles may be associated with both different growing regions and inherent genetic differences between different varieties of the same species. Additionally, previous research has demonstrated that different portions (cones, needles, limbs, trunk, etc.) of the same plant possess essential oils with varying volatile profiles [17-19].

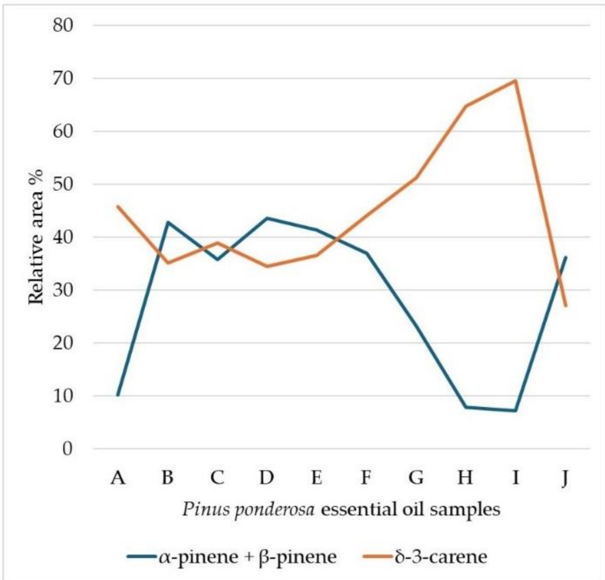

Figure 2. Line graph displaying inverse relationship between pinenes (sum of α- and β-pinene) and δ-3-carene.

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5. Conclusions

Basic plant chemistry research is critical in determining the chemical profiles of resins and is the initial step in understanding their potential future utility in the industrial, personal care, and/or medicinal fields.

The current study established the yield and essential oil profile of hydrodistilled resin collected from individual *Pinus ponderosa* var. *ponderosa* trees within a single native population. As expected within a population that propagates through seed dispersal, the chemical profiles of the naturally exuded resins were variable. This variability is likely further influenced by the state and age of the starting materials, namely naturally exuded resins. The current research provides foundational data on the potential of this novel natural product.

Authors' contributions

Conceptualization, data curation, formal analysis (GC/MS), methodology, sample procurement, software, validation, writing – original draft, T.M.W.; conceptualization, sample procurement, writing – original draft, writing – review and editing, C.P.; data curation, formal analysis (GC/MS), writing – review and editing, I.P.L.;
funding acquisition, validation, writing – review and editing, C.R.B.

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Availability of data and materials

All data resulting from this study have been included herein. Limited samples of the resin essential oils are available from the authors upon request.

Conflicts of interest

The authors declare no conflicts of interest. The funding entity had no role in the design of the study, nor in the collection, analysis, or interpretation of data.

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