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Chemical Composition of the Essential Oil and Supercritical CO_2 Extract of *Commiphora myrrha* (Nees) Engl. and of *Acorus calamus* L.

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Volatile concentrates from the oleo-gum resin of *Commiphora myrrha* (Nees) Engl. and from the rhizomes of *Acorus calamus* were isolated by supercritical extraction with carbon dioxide. The volatile oil of myrrh was obtained at 9.0 MPa and 50 °C and at a CO_2 flow of 1.5 kg/h. *Acorus calamus* was extracted at 9.0 MPa and 45 °C and at a CO_2 flow of 1.6 kg/h. In both cases, an oil devoid of cuticular waxes was obtained with a single depressurization stage. The SFE myrrh oil had a yield, *Y*, of 3.2%. Its main components, identified and quantified by GC/MS, were furanoeudesma-1,3-diene, 34.9%; lindestrene, 12.9%; curzerene, 8.5%; and germacrone, 5.8%. The essential oils from the same starting material by hydrodistillation, HD, ($Y = 2.8\%$) and by steam distillation, SD, ($Y = 0.4\%$) were quite similar to the SFE extract. The main components of the SFE oil of *A. calamus* ($Y = 3.5\%$) were acorenone, 13.4%; iso-acorone, 11.6%; (*Z*)-sesquilavandulol, 11.0%; dehydroxy isocalamendiol, 7.7%; and β-asarone, 5.5%. The comparison with hydrodistilled ($Y = 1.8\%$) and steam distilled ($Y = 1.0\%$) oils revealed large differences in the content of iso-acorone and crypto-acorone.

KEYWORDS: Supercritical carbon dioxide; essential oil; *Commiphora myrrha*; *Acorus calamus*

INTRODUCTION

Commiphora myrrha (Nees) Engl., known also as *Commiphora molmol*, belongs to the Burseraceae family. The genus *Commiphora* comprises over 150 species (*1*). The resin product widely known as myrrh is important both culturally and commercially. It is obtained as an exudate of these species, which are found abundantly in the dry arid regions of Ethiopia and Somalia (the largest producers and exporters of myrrh) and to a lesser extent in Kenya (*2*). The characteristic constituents of myrrh oil are furanosesquiterpenes such as furanoelemanes, furanoeudesmanes, and furanogermacranes (*3*). Indeed, a mixture of furanoeudesma-1,3-diene and lindestrene has the typical aroma of myrrh. Myrrh essential oil has been reported to have antiinflammatory (*4*) and antischistosomal (*5*) activity. Myrrh essential oil is not toxic (*6*) and is used to flavor cosmetics such as toothpaste and mouthwash, drinks, soft drinks, and food. This oil is generally obtained by hydrodistillation, steam distillation, and solvent extraction. Recent papers have reported the composition of the essential oil derived from the hydrodistillation of myrrh (*2*−*3*). Many works (*7*−*12*) have also been devoted to the identification or characterization of naturally occurring compounds in this oil and in oils obtained from other *Commiphora* species.

Acorus calamus L. (Araceae) is a native plant of India but grows also in the temperate zones of Europe, East Asia, and North America. In India, *A. calamus* and the essential oil of its rhizome serve mainly as an insecticide and insect repellent. Indeed, the rhizomes of Indian *A. calamus* possess insecticidal, larvicidal, antitermite, and larva and insect-repellant properties. In Ayurvedic medicine, the rhizomes are considered to possess antispasmodic, carminative, and anthelmintic properties and are used to cure many disorders, such as epilepsy, and mental diseases (*13*). The rhizomes of the European *A. calamus* and their essential oil are greatly used in the flavoring industry. The uses in Europe and India are different because the composition of the essential oil produced in the rhizomes of the respective plants is different (*14*). The phenylpropanoid, β-asarone, is dominant in the Indian oil from calamus rhizomes, while it is found in lower percentages in the European oils and in those from temperate regions in India, such as Kashmir (*15*). β-Asarone shows sedative and hypothermic but not analgesic effects (*16*). This compound has also been ascribed the carcinogenic properties of Indian calamus oil (*17*). This is the reason it has been banned in the United States as a food addictive. Recently, Sinha et al. (*18*) published a method to convert β-asarone into the nontoxic γ-asarone. The compositions of the essential oil from *A. calamus* rhizomes, obtained by hydrodistillation or solvent extraction, are reported in the literature (*13*−*15*, *19*−*21*). The variability in the composition of calamus essential oil has been ascribed to the existence of chemotypes with different ploidity (*20*). Four types have been characterized: diploid (β-asarone = 0%, North America), triploid (β-asarone in the

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Table 1. Chromatographic Area Percentages of the Four Main Constituents of the Fractions of Volatile Oil of *C. myrrha*[a]

compound	SFE-1	SFE-2	SFE-3	SFE-4	SFE-5
curzerene	9.7	11.5	9.6	9.5	10.6
furanoeudesma-1,3-diene	26.2	33.8	26.0	29.1	29.3
lindestrene	10.0	13.5	10.7	12.2	12.6
germacrone	2.8	4.1	4.3	5.1	4.8

[a] Obtained by SFE at 9.0 MPa and 50 °C, after each hour of extraction (SFE-1–SFE-5).

5−20% range, Europe), tetraploid (β-asarone up to 96%, East Asia, India, and Japan), and hexaploid (β-asarone $\approx$ 5%, Kashmir Region, India).

Extraction by carbon dioxide in the supercritical state is a process that offers many advantages in obtaining volatile extracts or aroma substances for human nutrition and for the pharmaceutical and perfume industries. Indeed, the mild extraction conditions give assurance against chemical reactions not taking place during the process. In particular, the SFE extracts are devoid of "burnt" notes characteristic of hydro- or steam-distilled oils. No study on obtaining volatile oil from the exudates of *C. myrrha* and from rhizomes of *A. calamus* by SFE is reported in the literature. The objectives of this study were to produce volatile concentrates from exudates of *C. myrrha* and from rhizomes of *A. calamus* by SFE and to compare them with oils obtained by conventional processes of extraction.

MATERIALS AND METHODS

Materials. Vegetable matter in both cases had been collected from the wild in 2002 and marketed by Minardi (Bagnacavallo-Ravenna, Italy). The exudates of *C. myrrha* come from Ethiopia, lot number F-050903100903. Unpeeled *A. calamus* L. rhizomes were gathered in the Haryana state, India, lot number F-170603170603. The water content, on dry basis, was 10.4% for *C. myrrha* and 5.7% for *A. calamus*. Before utilization, the material was ground with a Malavasi mill (Bologna, Italy) while care was taken to avoid overheating. In both cases, the particles size was in the range 250−850 μm. CO_2 (purity 99%) was supplied by SIO (Società Italiana Ossigeno, Cagliari, Italy).

SFE Apparatus. The supercritical CO_2 extractions were performed in a laboratory apparatus equipped with a 320 cm^3 extraction vessel operating in the single-pass CO_2 mode, through a fixed bed of ground material 20 cm high. About 300 g of myrrh or 250 g of calamus was charged at each run. A single separator allowed discharge of the essential oil at the desired time intervals. In this section, the temperature was maintained at a fixed value by means of a water thermostated system and using a heating ribbon wrapped around the pipe exiting the separator. A high-pressure diaphragm pump, Lewa model EL 1, with a maximum capacity of 6 kg/h, pumped liquid CO_2 at the desired flow rate. CO_2 was then heated to the extraction temperature in a thermostated oven controlled by a PID controller, model 2116 (Eurotherm). Extractions were carried out in a semibatch mode: batch charging of vegetable matter and continuous flow solvent. The carbon dioxide flow was monitored by a calibrated rotameter (Sho-rate, model 1355) located after the separator. Total CO_2 delivered during a run was measured by a dry test meter. Temperatures and pressures along the extraction apparatus were measured by thermocouple and Bourdon-tube test gauges, respectively. The pressure was regulated by high-pressure valves under manual control. Experiments were carried out at different conditions. No repeated runs were carried out.

Hydrodistillation. Hydrodistillations were performed for 4 h in a circulatory Clevenger-type apparatus up to exhaustion of the oil contained in the matrix, the same material used in the SFE. About 150 g of myrrh or 100 g of calamus was charged. No duplicate distillations were performed.

Steam Distillation. Steam distillations were performed in an Albrigi Luigi (Stallavena di Grezzana-Verona, Italy) stainless steel laboratory apparatus. Steam was generated from water placed at the bottom of the vessel (capacity 12 L) heated by direct fire. The ground vegetable material (about 350 g) was put on a perforated tray above the water level. The condensed water was recycled to the boiler. No duplicate distillations were performed.

Table 2. Kovats' Indices, I_K, and Chromatographic Area Percentages of Compounds Identified in the Volatile Extracts of the Oleo-Gum Resin of *C. myrrha* Obtained by Different Techniques[a]

compound	I_K	SFE	HD	SD
δ-elemene	1313	0.8	1.0	0.8
α-copaene	1353	0.3		0.3
β-bourbonene	1360	0.6	0.8	0.5
β-elemene	1367	3.0	4.3	3.6
E-caryophyllene	1392	0.6	0.5	0.6
γ-elemene	1403	0.3	1.1	0.6
α-humulene	1428	0.2		0.3
NI[b]	1443			0.3
γ-himachalene	1447			0.5
γ-muurolene	1449	0.4		0.6
germacrene D	1454	1.1	1.0	1.3
valencene	1485	1.0	0.9	1.3
curzerene	1470	8.5	17.5	14.7
NI	1472	0.9		
germacrene A	1479	0.8	0.4	0.8
δ-cadinene	1490			0.5
elemol	1520			0.2
germacrene B	1527	2.3	1.9	1.9
NI	1545	0.5	0.6	0.4
curzerenone	1568	1.4	0.9	1.2
furanoeudesma-1,3-diene	1590	34.9	38.6	33.5
lindestrene	1596	12.9	14.4	13.1
6-hydroxy-isobornyl isobutyrate	1602	3.5	3.8	3.0
NI	1617			1.1
atractylone	1621	2.8	2.5	1.7
8-hydroxy-isobornyl isobutyrate	1633	3.6	2.0	1.8
NI	1652	1.3	1.0	1.0
germacrone	1658	5.8	2.8	3.4
NI	1682	4.8	4.0	5.8
NI	1734			0.6
β-eudesmol acetate	1749	0.7		0.5
NI	1780	0.7		
NI	1787	0.5		
occidol	1794	0.7		0.4
benzyl salicylate	1827	0.6		0.2
NI	1849	2.7		2.3
NI	1866	0.2		
NI	1894	0.6		0.9
NI	1933	0.2		
NI	1961	0.5		
NI	1997	0.3		
NI	2160			0.6
total identified		86.8	94.6	87.4

[a] SFE at 9.0 MPa and 50 °C, SFE; hydrodistillation, HD, and steam distillation, SD. [b] NI, unidentified compound.

GC/MS Analysis. A Hewlett-Packard (Palo Alto, U.S.) 5890 series II gas chromatograph, GC, was employed. It was equipped with a split-splitless injector and a DB5-MS fused silica column; 5% phenylmethylpolysiloxane, 30 m × 0.25 mm i.d., film thickness 0.25 μm. The used GC conditions were programmed heating from 60 to 280 °C at 3 °C/min followed by 30 min under isothermal conditions. The GC was fitted with a quadrupole mass spectrometer, MS, model HP 5989 A. The injector was maintained at 250 °C. Helium was the carrier gas at 1.0 mL/min; the sample (1 μL) was injected in the split mode (1:20). The GC conditions were as follows: ionization energy 70 eV, electronic impact ion source temperature 200 °C, quadrupole temperature 100 °C, scan rate 1.6 scan/s, mass range (40−500) u. The software adopted to handle mass spectra and chromatograms was ChemStation. NIST98 (*23*), FLAVOUR, and LIBR(TP) (*24*) mass spectra libraries were used as references. Samples were run diluted in chloroform with a dilution ratio of 1:100. **Tables 1−3** show the chromatographic results, expressed as area percentages calculated without any response factor as a function of Kovats' indices, I_K (*25*). Identifications were made by

Table 3. Kovats' Indices, I_K, and Chromatographic Area Percentages of Compounds Identified in the Volatile Extracts of *A. calamus* Obtained by Different Techniques[a]

compound	I_K	SFE	HD	SD	compound	I_K	SFE	HD	SD
tricyclene	906			tr[c]	NI	1520		0.3	
α-pinene	916	tr	tr	0.3	*cis*-sesquisabinene hydrate	1524		0.5	0.6
camphene	933	0.2	0.2	1.6	β-calacorene	1532	0.4	0.4	0.5
sabinene	958			0.1	(*E*)-nerolidol	1536	0.6	0.8	0.7
o-cymene	1001			tr	NI	1539			0.4
sylvestrene	1006			0.2	spathulenol	1545	0.9	1.9	1.2
linalool	1072			0.5	caryophyllene oxide	1549	0.3	1.4	1.0
camphor	1112	0.6	1.2	1.0	(*Z*)-sesquilavandulol	1574	11.0	13.0	14.7
bornyl acetate	1263	tr	tr	0.2	NI	1574		2.0	
α-cubebene	1325	tr	tr	tr	tetradecanal	1576		1.0	2.6
α-copaene	1354	tr	tr	0.2	β-asarone	1583	5.5	5.1	4.7
NI[b]	1361	tr	tr	0.3	NI	1587		1.0	
β-elemene	1368	tr	0.2	0.4	dehydroxy isocalamendiol[d]	1593	7.7	3.5	3.2
1,7-diepi-α-cedrene	1380			0.2	NI	1598	0.9		
longifolene	1386		1.2	0.1	NI	1605	3.1	4.1	2.9
1,7-diepi-β-cedrene	1392	1.1		3.8	NI	1620	0.9		
β-cedrene	1396	0.4	0.4	1.3	selin-11-en-4-α-ol	1621	0.7	2.8	1.3
NI	1399	0.5		0.4	NI	1626	0.9	1.1	0.6
NI	1400	0.4			NI	1633	1.3	2.4	1.3
β-gurjunene	1403	1.0	0.9	2.7	cadalene	1637	1.0	1.0	0.5
NI	1406			0.2	NI	1643	0.7	1.1	0.4
trans-α-bergamotene	1409	0.4	0.4	1.2	α-asarone	1645	1.0		
aromadendrene	1424	0.5	0.4	1.1	NI	1645		1.8	0.5
α-himachalene	1429	1.1	1.1	2.6	acorenone	1655	13.4	21.6	11.5
allo-aromadendrene	1432			0.6	NI	1688	0.6	0.9	0.2
NI	1437			0.4	oplopanone	1703	1.2		
β-acoradiene	1440	tr	0.3	0.6	NI	1714	2.2	4.6	1.4
γ-curcumene	1449	0.4	0.6	1.2	aristolone	1723	0.8	1.6	
ar-curcumene	1459			0.6	NI	1756	1.4	0.9	
cis-β-guaiene	1462			1.5	*iso*-acorone	1771	11.6	1.4	0.3
shyobunone isomer[d]	1465	3.3	2.5	2.9	acorone	1775	2.0		0.7
α-selinene	1470	1.4	1.8	3.9	NI	1780	2.7		
trans-β-guaiene	1473			0.4	*crypto*-acorone	1792	5.0		
Z-α-bisabolene	1477	0.2			NI	1808	2.3		
shyobunone[d]	1487	2.6	7.0	7.8	NI	1811	1.0		
NI	1490	0.3		0.6	NI	1828	0.7		
δ-cadinene	1492	0.4	0.7	1.7	NI	2046			0.2
NI	1499	1.9	3.8	5.8	NI	2050			0.1
trans-calamene	1506		tr						
α-calacorene	1512	1.4	1.1	2.0	total identified		78.1	75.7	84.4

[a] SFE at 9.0 bar and 45 °C, SFE; hydrodistillation, HD; and steam distillation, SD. [b] NI, Unidentified compound. [c] tr, trace compound. [d] Compound identified by comparison of the only MS.

matching their mass spectra and I_K with those reported in the literature. **Table 3** also includes some compounds (denoted by [d]) that have been identified only on the basis of the mass spectra, the retention time referring to a DB5-MS fused silica column being unknown.

RESULTS AND DISCUSSION

Operative extraction conditions were chosen on the basis of previous results (*26*−*28*) on SFE of similar matrixes. The essential oils of the two species under investigation were obtained using a single depressurization stage because the starting materials were deprived of long-chain hydrocarbons. Separation conditions allowed the release of the oil and minimized the loss of fragrant compounds.

C. myrrha. In the SFE process, extraction pressure and temperature were set to 9.0 MPa and 50 °C, respectively. The solvent flow was 1.5 kg/h. The volatile concentrate of a pale yellow color was recovered in the separator that worked at 2.5 MPa and 25 °C. We performed a preliminary run drawing the extract in separate vials after each hour of extraction for 5 h. The components of myrrh volatile concentrate can be grouped into two classes: hydrocarbon sesquiterpenes, HS, and oxygenated sesquiterpenes, OS, on the basis of the chemical structure or the retention times for nonterpenoids. Indeed, hydrocarbon and oxygenated monoterpenes are absent in this oil. The dependence of the percentage of the different classes of terpenes on extraction time is very weak; HS vary from 24 to 15% and OS are in the range 76−84%. This is confirmed by the values of the percentages of the main constituents in each sample collected, in decreasing amount, after every hour of extraction as reported in **Table 1**. This trend is indicative of the great availability of essential oil constituents in the starting material. A similar behavior was observed in the case of SFE of the essential oil from clove buds (*28*). On the contrary, when the compounds of interest show strong affinity toward the vegetable structure, the relative amount of a compound or of a particular class of compounds strongly depends on the extraction time (*29*−*30*). The SFE column of **Table 2** shows the analytical results from the sample obtained by putting all fractions in a single vial from a run at the above-mentioned conditions. Twenty-six compounds were identified amounting to 86.8, 94.6, and 87.4% in the SFE, HD, and SD extracts, respectively. The corresponding yield expressed as percent w/w, compared to the charged material, *Y*, was 3.2%. The same table also shows the composition of the oils obtained by hydrodistillation, HD, ($Y = 2.8\%$) and steam distillation, SD, ($Y = 0.4\%$). The very low yield of the SD oil can be attributed to the fact that the collecting device is not suitable to recover oils such as myrrh oil that have

a density higher than that of water. The extract obtained by SFE contained mainly furanoeudesma-1,3-diene, 34.9%; lindestrene, 12.9%; curzerene, 8.5%; germacrone, 5.8%; an unidentified compound having $I_K = 1682$, 4.8%; 8-hydroxy-isobornyl isobutyrate, 3.6%; 6-hydroxy-isobornyl isobutyrate, 3.5%; and β-elemene, 3.0%. A comparison with the hydrodistilled oil (HD) did not reveal any large differences, except for curzerene, 8.5% versus 17.5%, and germacrone, 5.8% versus 2.8%, in the SFE and the HD sample, respectively. The oil obtained by SD was very similar to the hydrodistilled oil and to a lesser extent to that obtained by SFE.

No paper concerning SFE of this essential oil has been found in the literature. Our results are in good agreement with those found by Baser et al. (*2*), who obtained an HD myrrh oil made up of the following main components: furanoeudesma-1,3-diene (34.0%), furanodiene (19.7%), and lindestrene (12.0%). Morteza-Semnani and Saeedi (*3*) published the composition of the essential oil of *C. myrrha* var. *molmol* from Iran produced at a yield (w/w) of 3.1% by HD. They identified 32 constituents, including in decreasing order curzerene (40.1%), furanoeudesma-1,3-diene (15.0%), and β-elemene (8.4%).

A. calamus. On a charge of ground rhizomes of calamus, the volatile oil was isolated with supercritical CO_2 at 1.6 kg/h and 9.0 MPa and 45 °C in the extraction section. The pressure and temperature in the separator were 2.0 MPa and 28 °C. The process was extended up to 8 h. We compared the *A. calamus* oils obtained with three different methods: supercritical extraction, SFE; hydrodistillation, HD; and steam distillation, SD. **Table 3** shows the detailed identification and area percentages of the 50 compounds found in the oils. The main compounds responsible for the fragrance of the *A. calamus* oils obtained by SFE, HD, and SD are, respectively, acorenone, 13.4% versus 21.6% versus 11.5%; iso-acorone, 11.6% versus 1.4% versus 0.3%; (*Z*)-sesquilavandulol, 11.0% versus 13.0% versus 14.7%; dehydroxy isocalamendiol, 7.7% versus 3.5% versus 3.2%; β-asarone, 5.5% versus 5.1% versus 4.7%; cryptoacorone, 5.0% versus 0% versus 0%; shyobunone, 2.7% versus 7.0% versus 7.8%; and acorone, 2.0% versus 0% versus 0.7%. No dramatic differences were observed in the different samples. The oil obtained by steam distillation contains a higher percentage of hydrocarbon monoterpenes (2.2%), oxygenated monoterpenes (1.5%), and hydrocarbon sesquiterpenes (26.6%). In contrast, the SFE oil and the HD oil are richer in oxygenated sesquiterpenes than the SD oil: 62.1, 59.0, and 46.4%, respectively.

Thanks to supercritical extraction, it was possible to avoid the partial hydrolysis of iso-acorone, acorone, and cryptoacorone which occurs when water is used as a distillation medium. Indeed, the solubility of these acorones in water is not negligible, and traces of these compounds were revealed in the aqueous medium that came into contact with the oils during HD and SD extraction. This experimental evidence confirms that SFE avoids the degradation of thermolabile compounds, hydrolysis phenomena, and the solubilization of water-soluble compounds. The three techniques differ also with respect to the yields of extraction: the best yield, 3.5 wt % of the charged material, was obtained in SFE versus 1.8% in HD and 1.0% in SD. A calamus oil with a nondissimilar composition was obtained by HD from Lithuanian rhizomes at a yield of 1.2% by Venskutonis and Dangilyte (*21*). As main components, they found acorenone, 20.9%, isocalamendiol, 12.8%, and shyobunone isomer, 7.8%. The β-asarone content was 2.3%. Thirty-one constituents, among the 55 identified, are also present in our volatile extracts. Raina et al. (*13*) identified β-asarone (83.2%) and α-asarone (9.7%) as major constituents in the rhizome oil of *A. calamus* from the lower region of the Himalayas. Mazza (*22*) compared the calamus essential oil from two different sites. β-Asarone was present as 77.68% and 5.24% in the Indian and in European oil, respectively.

Since our sample was from the state of Haryana, North India, it is more similar to other samples from Europe and the Kashmir region (*15*) rather than from other Indian sites.

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