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Yield and Chemical Composition of the Essential Oil of the Stems and Rhizomes of *Cyperus articulatus* L. Cultivated in the State of Pará, Brazil

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Abstract

The volatile constituents from the stems and rhizomes of *Cyperus articulatus*, cultivated in the state of Pará, Brazil, were obtained by hydrodistillation and analyzed by GC and GC/MS. The major components of these oils were α-pinene (0.7-12.9%), mustakone (7.3-14.5%) and caryophyllene oxide (4.6-28.5%). Maximum yield in essential oil was furnished by the rhizomes hydrodistilled at 5 h.

Key Word Index

Cyperus articulatus, Cyperaceae, essential oil composition, α-pinene, mustakone, caryophyllene oxide.

Introduction

The genus *Cyperus* L., belonging to the family Cyperaceae, which consists of about 550 species, are generally grass-like in appearance and are usually found in marshy or aquatic habitats (1). Essential oils from *Cyperus* species are generally constituted mostly of sesquiterpenoids, and minor monoterpenoids (2-4). *Cyperus articulatus* L. [syn. *Chlorocyperus articulatus* Rikli, *Cyperus articulatus* var. *multiflorus* Kük., *C. articulatus* var. *nodosus* (Humb. et Bonpl. ex Willd.) Kük., *C. corymbosus* Rottb., *C. corymbosus* var. *subnodosus* (Nees et Meyer) Kük., *C. diphyllus* Pretz., *C. niloticus* Forssk., *C. nodosus* Humb. et Bonpl. ex Willd., *C. nodosus* var. *subnodosus* (Nees et Meyer) Boeck, *C. subnodosus* Nees et Meyen] is a tropical sedge widely distributed in the Amazonian region, where it is commonly known as "piripiri."

In the state of Pará, *C. articulatus* is known as "priprioca" or "piriprioca," the rhizomes forming tubers, which possess a very pleasant and strong smell. A hexane extract of *C. articulatus* has already been investigated by many groups and several sesquiterpenes have been isolated (5-9). In the Amazonian region, "piripiri" has been used to treat many diseases (10). The decoction of the *C. articulatus* has been found to be effective against *Pseudomonas aeruginosa* and *Staphylococus aureus* (11). Water extracts of their rhizomes were reported to be active to epileptiform dischages (12). In the state of Pará, the most widely cultivated of the *Cyperus* species were *C. articulatus*, *C. compressus* and *C. prolyxus*, locally known as "priprioca," "priprioquinha" and "pripriocão," respectively. These plants are cultivated in the home gardens ("quintal") to treat dysentery and headache. *Ciperus articulatus* is also cultivated by small holders, and sold in open markets in the city of Belém, mostly in Ver-o-Peso, and it is used by the local cosmetic industries. As a part of the development of the our research about the economic production of the aromatic plants in the state of Pará, this paper relate the yield and the chemical composition of the essential oil of the stems and rhizomes of *C. articulatus*.

Experimental

Material and distillation of the volatile constituents: Sample A was obtained from the Ver-o-Peso open market, in the city of Belém, March 2003. Sample B was obtained from the small holders at the site of cultivation, and harvested for eight months (Municipality of Acará), July 2002. Samples C and D were collected from a home garden in the municipality of Augusto Corrêa, March 2003. Sample E was collected from a home garden in the municipality of Bragança, March 2003. Samples B, C, D and E were collected at their flowering stage. Voucher specimens (B: 167612, C: 168,788, D: 168794, E: 168783) have been deposited in the Herbarium of Museu Paraense Emílio Goeldi. The samples were dried for seven days in a room under in an air-conditioned environment, and ground when dry. The oils from each sample (Sample A: 200.0 g; Sample B: 507.2 g; Sample C: 80.0 g; Sample D_1: 52.0 g; Sample D_2: 26.0 g; Sample E_1: 74.0 g; Sample E_2: 50.0 g) were obtained by separate hydrodistillation for 3 h, using a Clevenger-type apparatus. The residual humidity was determined

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Table I. Components (%) of the stem and rhizome oils of *Cyperus articulatus*

Components	RI*	Sample A	Sample B	Sample C	Sample D_1	Sample D_2	Sample E_1	Sample E_2
α-pinene	936	12.9	12.3	7.7	5.7	0.9	6.5	0.7
camphene	953	0.2		0.1	0.1			
thuja-2,4(10)-diene	955	1.2	0.9	0.4	0.7		0.7	
sabinene	976	0.4		0.2	0.1	0.4		0.7
β-pinene	980	7.4	6.6	7.4	4.2	0.7	4.4	0.4
p-cymene	1025	0.6	0.8	0.2	0.3	0.2	0.3	0.2
limonene	1030	1.2	1.3	0.6	0.7	3.6	0.7	4.1
1,8-cineole	1032	0.3		0.8	0.2		0.2	
p-cymenene	1089	0.4		0.4	0.4		0.3	
α-campholenal	1126	0.7	0.7	0.3	0.6	0.7	0.4	0.1
trans-pinocarveol	1138	7.5	5.9	3.8	6.0	0.2	5.5	0.4
trans-verbenol	1144	1.7	1.6	1.4	2.7	0.1	2.4	0.2
cis-verbenol	1140	1.2	1.0	0.3	0.4		0.4	
pinocarvone	1166	1.9	1.6	1.6	2.1	0.1	1.4	0.2
p-mentha-1,5-dien-8-ol	1170	3.0	2.8	0.8	1.2		1.2	
terpinen-4-ol	1177	0.8	1.0	0.2	0.2		0.2	0.2
p-cymen-8-ol	1185	0.8		0.3	0.6		0.5	
myrtenal + myrtenol	1195	7.7	6.3	4.1	5.9	0.3	5.8	0.5
verbenone	1206	2.6	3.9	1.1	2.1		1.5	
trans-carveol	1217	0.6	0.6	0.2	0.6	0.2	0.5	0.7
cis-carveol	1229	0.1		0.1	0.1		0.1	0.2
carvone	1242	0.5	0.5	0.2	0.4	0.4	0.2	1.0
p-cymen-7-ol	1287	0.2		0.1	0.1	0.5	0.1	
cypera-2,4-diene	1360	0.1			0.3		0.3	
α-copaene	1374	2.0	3.3	2.1	2.2	2.1	2.3	2.5
β-elemene	1392	0.1		0.3	0.1			0.2
cyperene	1396	1.1	1.8	0.6	4.3	1.1	3.2	1.2
β-caryophyllene	1418			0.7	0.1			0.2
α-guaiene	1439	0.3		0.6	0.2	0.3	0.2	0.1
α-humulene	1454	0.1		1.5	0.2	0.6	0.2	1.7
rotundene	1457	0.5	0.5	0.4	0.8	0.4	0.8	0.4
germacrene D	1485	0.6		0.7	0.9	1.0	1.0	1.9
eudesma-2,4,11-triene	1469	0.9	0.7	0.9				
β-selinene	1488	1.9	2.7	2.0	0.4	0.3	0.3	1.8
α-selinene	1495	1.2	1.2	1.7	0.2	1.0		2.1
α-bulnesene	1508	1.0	2.1	1.8	0.6	0.9	0.6	0.7
δ-cadinene	1522	0.7	0.8	0.9	0.4	1.8	1.0	1.6
trans-calamenene	1529	0.3	0.5	0.3	0.8	0.2	0.5	0.4
α-calacorene	1544	0.7	0.8	0.7	1.1	2.1	1.4	2.5
ledol	1565	4.3	3.2	4.0	4.3	7.3	4.6	8.8
caryophyllene oxide	1580	7.0	4.6	13.7	10.8	27.4	10.1	23.3
humulene epoxide II	1605	1.4	1.3	11.2	2.4	6.5	2.4	5.5
β-copaen-4α-ol	1590	0.3		0.7	0.5	0.5	1.0	0.4
dillapiole	1620	0.8	1.0		1.3	5.3	1.4	4.5
patchoulenone	1616	0.5	1.0	0.4	1.6	1.4	1.5	1.4
caryophylla-4(14),8(15)-dien-5α-ol	1641				0.5	1.1	0.6	0.7
M 218	1643		2.1	1.4	3.6	6.3	4.4	6.1
eudesma-3,11-dien-5-ol	1633	0.8		1.6	0.7			
mustakone	1676	7.3	9.8	8.0	12.9	8.2	14.5	10.0
cyperotundone	1694	2.1	4.5	3.7	3.7	6.7	5.4	6.3
M 218	1705	2.6	0.5	1.5	1.5	4.3	1.3	4.6
M 220	1740	0.8	0.5	0.6	0.6	1.3	0.7	0.9
α-cyperone	1755	5.4	5.9	4.2	5.2	1.3	1.4	0.7
aristolone	1763	0.5	0.5	0.6	0.6	0.4	0.6	0.5

*RI on DB5-MS; Samples A, B, C, D_1 and E_1 = rhizome; Samples D_2 and E_2 = stem; RI=1643, m/z (rel. int.): 218[M+] (4), 203(7), 189(11), 175(26), 161(36), 147(29), 133(46), 121(67), 107(77), 93(100), 91(92), 71(53), 67(47), 55(59), 43(96); RI=1705, m/z (rel. int.): 218[M+](57), 203(67),189(10),175(34), 161(60),147(50), 133(46), 119(60),105(72), 91(100), 79(50), 67(48), 41(53); RI=1740, m/z (rel.int.): 220 [M+](9), 205(29), 187(16), 159(32), 145(29), 133(49), 119(48), 107(87), 105(100), 93(71), 91(69), 79(60), 67(64), 55(43), 43(40)

by azeotropic distillation using toluene and Dean & Stark collector (13). The oils obtained were dried over anhydrous sodium sulfate, and the samples immediately submitted to GC/FID and GC/MS analysis.

GC/MS: GC/MS was performed on a Finnigan Mat INCOS XL GC/MS system, equipped with a DB-5MS (30 m x 0.25 mm, 0.25 µm film thickness) fused silica capillary column; the carrier gas was helium adjusted to a linear velocity of 32 cm/s (measured at 100°C); split flow was adjusted to give a 20:1 ratio, and septum sweep was a constant 10 mL/min. Splitless injection of 1 µL, of a 2:1000 hexane solution; injector and detector temperature was 250°C; oven temperature programmed was 60°-240°C at

3°C/min. EIMS: electron energy, 70 eV; ion source temperature and connection parts: 180°C. Individual components were identified by comparison of both mass spectrum and their GC retention data with those of authentic compounds previously analyzed and stored in the data system, and by comparison of mass spectra with those in the data system libraries and cited in the literature (3,14).

GC: Analysis of volatile components were performed on a HP5890-II instrument, in the same conditions as above except of the hydrogen as carrier gas. The GC was equipped with FID and connected with an eletronic integrator HP 3396 Series II. The percentage composition of the oil samples were computed from the GC peak areas without using correction for response factors. Mustakone was isolated from a hexane extract by repeated column chromatography over silica gel, and its identification was assigned on the basis of spectroscopic data (NMR Spectrometer Gemini 300/Varian, 300 and 75MHz, $CDCl_3$), which were previously described (4).

Results and Discussion

The composition of the oils and retention indices are given in Table I. The rhizome oils of *C. articulatus* were strongly yellow and furnished a yield of 0.5-1.0% for the samples contained 35.4% of the residual humidity (yield calculated at dried basis were 0.9% to 1.6%, respectively). Maximum yield was obtained from 5 h of distillation. The stems were weak in oil and yielded only < 0.05%. The components of the oil of *C. articulatus* were the groups commonly occuring in oils of Cyperaceae, comprising monoterpenes and sesquiterpenes. The monoterpenes α-pinene, β-pinene, *trans*-pinocarveol, *trans*-verbenol, pinocarvone and myrtenal + myrtenol, were major in the rhizome oils, except of limonene, which showed a higher content in the stem oils. In the group of sesquiterpenes, ledol and caryophyllene oxide contents were higher in the stem oils. The content of mustakone was higher in the rhizome oils. Other constituents of the oils were typical of the Cyperaceae such as cyperene (0.6-4.3%), rotundene (0.4-0.8%%), cypera-2,4-diene (0.1-0.3%), patchoulenone (0.5-1.6%), cyperotundone (2.1-6.7%) and α-cyperone (0.7-5.9%). Cyperene and cyperotundone are common in other oils of Cyperaceae family, such as *Cyperus maculatus* Boëck, *C. rotundus* L., *Kyllinga erecta* S. and *Scleria striatinux* De Wild. (2,3,15,16). Mustakone was previously reported on the oils of *C. rotundus* (17,18), *C. maculatus* (2), and a hexane extract of *C. articulatus* (4). The chemical profile of the rhizome oil of *C. articulatus* was very similar to those reported for *C. maculatus* (2). The difference in the relative proportion of the components of the oils can be atributed to different growing conditions, perhaps to differing agronomic practices, and different harvesting time.

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