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ESSENTIAL OILS FROM BRAZILIAN *RUTACEAE*. I. GENUS *PILOCARPUS*

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Abstract.—Essential oil of *Pilocarpus microphyllus* Stapf, *P. jaborandi* Holmes, *P. affinis pauciflorus* Stapf. *P. sp* 192 and *P. sp* 212 contains terpenes, sesquiterpenes and ketones. Some of them are reported for the first time in the genus.

The leaves of jaborandi are commercially exploited in the Brazilian Northeast for industrial extraction of pilocarpine, an alkaloid with potent cholinergic activity (1).

In the leaves, there is also an essential oil present whose composition is registered in the literature in a confusing and incomplete way (2, 3, 4) which might be attributed to improper selection of plant material or lack of suitable identification because analyses were made on commercial materials. Therefore, chemical reexamination of the essential oil from five distinct *Pilocarpus* species was conducted together with an analysis of the leaves used by local industry.

DISCUSSION

The plants were collected in the states of Ceará, Maranhão, and Pernambuco. Commercial material was obtained from the industry in the state of Piauí. The last material was mainly composed of *Pilocarpus microphyllus* Stapf. leaflets according to pharmacognostic examination (3). *P. jaborandi* Holmes, *P. microphyllus* and *P. affinis pauciflorus* Stapf. were identified among the species collected; two other distinct species of the genus are in the stage of botanical identification. Essential oil from each sample was obtained by steam distillation. Yields and collection sites are described in table 1.

Table 1. Essential oils of pilocarpus species: yields and collections sites.

No.	Common name	Botanical species	From	Oil yield %
01	Jaborandi do Maranhão	*P. mycrophyllus* Stapf.	S. Quitéria—Ma. lat. long. 3°33'S 42°35'W	0.25
02	Jaborandi Branco	*P. jaborandi* Holmes	Meruoca—Ce. lat. long. 3°30'S 40°30'W	0.5
03	Jaborandi	*P. affinis pauciflorus* Stapf.	Crato—Ce. lat. long. 7°10'S 39°25'W	0.6
04	Jaborandi da Folha pequena	*Pilocarpus* sp.—192	Viçosa—Ce. lat. long. 9°28'S 36°25'W	1.2
05	Pimentinha	*Pilocarpus* sp.—212	Bodocó—Pe. lat. long. 7°45'S 39°55'W	0.6
06	Comercial jaborandi	*P. mycrophyllus*[a] Stapf.	Parnaíba—Pi. lat. long. 2°25'S 41°45'W	0.3

[a]Major species present in the mixture of leaflets.

Table 2 presents the chemical composition of the essential oil from the five *Pilocarpus* species and includes, also, analysis of a commercial sample. α-Pinene is the only constituent present in all species studied, and the rare diterpene sandaracopimaradiene is characteristic of *Pilocarpus jaborandi*.

The chemical constituents of the essential oil from commercial pilocarpus leaves confirms the pharmacognostic identification of *P. mycrophyllus* as the major species present in the mixture. The presence of all compounds except limonene and 2-undecanone is being reported for the first time in the genus.

TABLE 2. Chemical composition of essential oils from *Pilocarpus* species from Brazilian northeast.

	Component[c]	Species[a]						Identification process
		I	II	III	IV	V	VI	
1	limonene	47.0[b]	2.0	4.0	—	7.0	—	ir, pmr, ms
2	myrcene	0.5	—	2.0	—	4.0	—	ir, pmr, ms
3	β-ocimene[d]	—	1.0	—	—	—	—	ms
4	α-pinene	2.5	1.0	8.0	2.5	13.0	0.8	ir, pmr, ms
5	sabinene	—	—	20.0	6.0	30.0	3.0	ir, pmr, ms
6	α-terpinene	—	—	2.0	—	4.0	—	ir, pmr, ms
7	γ-terpinene	—	—	—	—	10.0	—	ir, pmr, ms
8	tricyclene[d]	—	—	—	—	1.0	—	ms
9	3,7,7-trimethylbicyclo[3.1.1.]-2-heptene[d]	—	12.0	2.0	—	—	—	ms
10	terpinen-4-ol[d]	—	—	—	—	1.0	—	ms
11	Δ-cadinene[d]	—	4.7[b]	—	—	11.0	5.6	ms
12	γ-cadinene[d]	—	—	—	—	11.0	—	ms
13	caryophyllene	—	19.0	—	8.0	5.0	15.0	ir, pmr, ms
14	α-copaene	—	3.0	—	—	—	9.5	ir, pmr, ms
15	γ-elemene	—	—	—	—	4.0	—	pmr, ms
16	α-guayene	—	4.4	—	—	—	—	ir, ms
17	humulene	—	4.0	—	—	1.0	—	ir, pmr, ms
18	β-selinene[d]	—	—	2.0	—	—	—	ms
19	sandaracopimaradiene	0.5	—	—	—	—	—	ms, pmr
20	2-undecanone[d]	22.5	—	43.0	33.0	—	—	ms
21	2-tridecanone[d]	—	22.0	—	—	—	23.0	ms
22	vinyl dodecanoate[d]	0.5	—	—	—	—	—	ms

[a]I=*Pilocarpus jaborandi;* II=*P. microphyllus;* III=*P. affinis pauciflorus;* IV=*Pilocarpus* sp. 0192; V=*Pilocarpus* sp. 0212; VI=Comercial jaborandi leaves.

[b]Calculated by glc.

[c]Authentic samples were supplied by Dragoco S.A. (Sao Paulo, Brasil) or from the author's collection.

[d]Comparison with reference spectrum (6). No authentic samples available.

EXPERIMENTAL

The plants were collected on the locations mentioned in table 1. Botanical identifications were made by Prof. Afrânio G. Fernandes. Voucher specimens were deposited in the herbarium of the Departamento de Biologia—Universidade Federal do Ceará under number 3023, 2969, 3229, 3251 and 3563.

ISOLATION OF CONSTITUENTS.—Essential oil extraction was carried out by steam distillation of finely ground leaves in an apparatus developed in our laboratory (5). The yields (volume/volume) are reported in table 1. Each essential oil, after separation, was dried with anhydrous sodium sulfate, filtered, and sealed in glass vials under nitrogen atmosphere.

The fresh oils were analyzed by glc on a VARIAN FID instrument equipped with a stainless steel column (1.5 m x 3.0 mm) packed with 5% OV-101 on chromosorb W, at the programmed temperature 50–250°C/4°/min, and with nitrogen as carrier gas.

Limonene, myrcene, α-pinene, sabinene, caryophyllene, humulene, γ-elemene, and sandaracopimaradiene were isolated by preparative glc performed in a VARIAN FID instrument with an aluminum column (6.5 m x 9 mm) packed with 3% OV-101 on chromosorb W.

The oils were submitted to gc/ms separation on a FINNIGAN 3300 quadrupole mass spectrometer coupled to a gc equipped with a glass column (1,5 m x 3,1 mm) packed with 3% OV-101 on chromosorb W with helium as the carrier gas and ms spectra recorded at 70 eV. The infrared spectra were obtained on a Zeiss UR-20 instrument using liquid films.

The pmr spectra were obtained on a VARIAN 60 MHz instrument in carbon tetrachloride solution with TMS as internal standard.

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