European Journal of Medicinal Plants

32(1): 37-45, 2021; Article no.EJMP.65734
ISSN: 2231-0894, NLM ID: 101583475

Oils Variability of *Pectis elongata* in the Amazon and an Overview of the Neotropical *Pectis* Species

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Authors' contributions

This work was carried out in collaboration among all authors. Authors LTM, RHVM and JGSM designed the study, performed the statistical analysis, wrote the protocol, and wrote the first and final drafts of the manuscript. Authors LTM, RHVM, CS and SLFS managed the analyses of the study. Authors LTM and JGSM managed the literature searches. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/EJMP/2021/v32i130362
Editor(s):
(1) Dr. Patrizia Diana, University of Palermo, Italy.
(2) Prof. Marcello Iriti, Milan State University, Italy.
Reviewers:
(1) Krishna Sujeethasai, University of Jaffna, Sri Lanka.
(2) Manuel Alejandro Melendez Jimenez, Médico Veterinario de la Universidad de la Salle, Colombia.
Complete Peer review History: http://www.sdiarticle4.com/review-history/65734

Original Research Article

Received 10 December 2020
Accepted 15 February 2021
Published 03 March 2021

ABSTRACT

Aims: *Pectis* species, belonging to Asteraceae, are aromatic and medicinal herbaceous plants, distributed in the Americas, the West Indies, and the Pacific islands, with lemon, cumin, or oregano aroma, used in infusions to treat several diseases or as spices.
Methodology: In this study, the composition of *Pectis elongata* from the Amazon was correlated with other Neotropical *Pectis* oils, including their traditional uses and biological properties. *Pectis elongata* oils were obtained by hydrodistillation and analyzed by GC and GC-MS.

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Results: Citral (39.1% of neral and 47.7% of geranial), perilla aldehyde (51.7% to 81.8%), and limonene (33.7% to 43.7%) were identified as their primary constituents of the *Pectis elongata* oils from the Amazon. It is assumed the existence of at least two chemotypes for the variability of the oils of *P. elongata*: Citral (neral plus geranial) and perilla aldehyde plus limonene. The main C10-skeletal monoterpenes found in *Pectis* oils can be depicted according to their biosynthetic pathways: neral and geranial arranged in an acyclic-type skeleton, and limonene, perilla aldehyde, cumin aldehyde, carvone, p-cymene, and thymol in a *p*-menthane-type skeleton.
Conclusion: There is a particular interest in the world's citral-rich essential oils, such as *Pectis elongata* oil and other similar *Pectis* oils, given their application in human health and food preservation.

Keywords: Asteraceae; essential oils; citral; perilla aldehyde; limonene; traditional uses and biological properties.

1. INTRODUCTION

Asteraceae, the sunflower family, with about 24,000 species, grouped into about 1,700 genera, is the largest among Angiosperms, representing 10% in the world, with cosmopolitan distribution and broad coverage in the temperate and semiarid regions of the tropics and subtropics. It comprises about 280 genera and 2,000 species in Brazil, occurring in different vegetation types [1].

Pectis L. is the largest genus of the marigold tribe (Pectidinae: Tageteae), comprising about 90 annual and perennial species, adapted to warm regions of the New World, and occurring in savannas and openings of dry tropical forests of North America, Mexico, West Indies, Central and South America, and Pacific Islands. It is also characterized by opposite leaves with pairs of bristles at their bases, adnate phyllaries, having a floret as a single unit at maturity, and concise and densely papillose style branches [2]. *Pectis* species have noticeable oil glands on the leaves' margins and undersides, yielding essential oils with intense spicy scents used in teas, spicing food, and medicinal purposes [3-9]. In general, these species display the scent of lemon, curry, tarragon, or fennel. Also, a few species have a disagreeable odor likened to that of bedbugs [10].

Pectis elongata Kunth [syn. *Pectis elongata* var. *floribunda* (A. Rich.) D. J. Keil, *P. floribunda* A. Rich., *P. elongata* var. *oerstediana* (Rydb.) D. J. Keil, *P. oerstediana* Rydb., P. *elongata* var. *fasciculiflora* (DC.) D. J. Keil, among others] [11], belongs to Asteraceae and has a wide geographical distribution, coming from Central America to Brazil, Colombia, Venezuela, and Guianas. It is an annual herb (15-50 cm), erect, with violet leaves and slightly purple yellow flowers, pointed and full of glandular oil, commonly known as "cuminho-bravo," and "limãozinho" in Pará and Amapá states, North Brazil, due to the scent of two different types, which resembles cumin (*Cuminum cyminum* L.) and citronella [*Cymbopogon citratus* (DC.) Stapf] [12].

Fig. 1. *Pectis elongata* Kunth

The present work aimed to extract and analyze the composition of the essential oil of a specimen of *Pectis elongata*, with occurrence in Santarém, state of Pará, Brazil, and carry out a literature revision concerning all data previously reported with *Pectis* species.

2. MATERIALS AND METHODS

2.1 Plant material and Collection Data

Pectis elongata (whole plant) was sampled in the city of Santarém, state of Pará, Brazil, during the dry season (October 2017), at a location with the following geographic coordinates: 02º27.8'143" S / 54º41.31'646" W. The plant was identified and

deposited in the Herbarium of Universidade Federal do Oeste do Pará, at Santarém, state of Pará, Brazil, under the number HSTM-003432. The botanical material was dried for three days at room temperature, ground, and then submitted to essential oil extraction.

2.2 Essential Oil Distillation

The essential oil was obtained by hydrodistillation, using a Clevenger-type apparatus (3 h), with three replicates. Oil was centrifuged with anhydrous sodium sulfate to remove residual water, stored in a labeled amber glass bottle, and kept under refrigeration at 5ºC. The oil yield was calculated by the plant biomass free from the moisture, utilizing the relationship between the volume of obtained oil and the dry biomass used in the extraction (v/w %). The essential oil sample was named PeSTM.

2.3 Oil Composition Analysis

The essential oil analysis was performed on a GCMS-QP2010 Ultra system (Shimadzu Corporation, Tokyo, Japan) equipped with an AOC-20i auto-injector and the GCMS-Solution software containing the Adams (2007), NIST (2011), and Mondello (2011) libraries [13-15]. A silica capillary column (Rxi-5ms, 30m x 0.25mm; 0.25µm film thickness, Restek Corporation, Bellefonte, PA, USA) was used. The conditions of analysis were: injector temperature of 250°C; oven temperature programming of 60-240°C (3°C/min); helium as the carrier gas, adjusted at a linear velocity of 36.5 cm/s (rate of 1.0 ml/min); injection of 1 µL of the sample in the split mode (5 µL of the essential oil to 500 µL of hexane); split ratio 1:20; ionization by electronic impact at 70 eV; the temperatures of ionization source and transfer line at 200 and 250°C, respectively. The mass spectra were obtained by automatic scanning every 0.3 s, with mass fragments in the range of 35-400 m/z. The retention index was calculated for all volatile components using a homologous series of C_8-C_{20} n-alkanes (Sigma-Aldrich), according to the linear equation of Van den Dool and Kratz (1963) [16]. The quantitative data regarding the volatile constituents were obtained by peak-area normalization using a Shimadzu GC 2010 ultra-system, coupled to FID Detector, operated under similar GC-MS system conditions. The constituents were identified by comparing their retention indices and mass spectra (molecular mass and fragmentation pattern) with those existing in the GCMS-Solution system libraries [13-15].

2.4 Literature Review

It was conducted regarding the widespread uses, properties, biological activities, and oils and extracts of other *Pectis* species' composition.

3. RESULTS AND DISCUSSION

The oil yield of *P. elongata* (PeSTM) sampled in Santarém, PA, Brazil, was 2.5%. GC-FID and GC-MS analysis have determined the quantification and identification of the PeSTM oil constituents, as shown in Table 1. Twenty-three constituents were identified, totalizing 98.6%. The predominant compound in the oil was citral (86.8%), represented by the sum of the two isomeric oxygenated monoterpenes, neral (39.1%) and geranial (47.7%). Other related monoterpenes, like limonene, linalool, citronellal, *exo*-isocitral, (*Z*)-isocitral, (*E*)-isocitral, nerol, and geraniol, were also found in minor amounts in the oil.

The oils of three other *Pectis elongata* samples, collected in the cities of Belém (PeBEL) and Bujaru (PeBUJ), Pará state, and the town of Ferreira Gomes (PeFEG), Amapá state, Brazil, were previously reported by us [17]. These oil compositions have now been revised using updated libraries [14,15]. The PeBEL and PeBUJ oils yields were 0.7% and 1.3%, respectively, for which seven volatile compounds were identified in PeBEL and six in PEBUJ, totaling 99.1% and 99.8% in both oils, having perilla aldehyde (51.7% and 64.6%) and limonene (43.7% and 33.7%) as its main constituents. The PeFEG oil yield was 1.5%, seven volatile compounds were identified in the oil, totalizing 92.3%, with perilla aldehyde (81.8%), perilla alcohol (5.6%), and perilla acid (4.0%) as its primary components (see Table 1).

In general, as seen, *Pectis* species have pleasant citrus, cumin, and oregano-like scents due to the presence of monoterpene constituents in their volatile compositions, as citral (neral plus geranial), α- and β-pinene, limonene, perilla aldehyde, cumin aldehyde, carvone, *p*-cymene, and thymol. The main C_{10}-skeletal monoterpenes found in the *P. elongata* oil and other *Pectis* oils can be depicted according to their biosynthetic pathways: (1) neral and geranial arranged in an acyclic-type skeleton, (2) α-pinene and β-pinene in a pinane-type skeleton, and (3) limonene, *cis*- and *trans*-limonene oxide, perilla aldehyde, cumin aldehyde, carvone, *p*-cymene, thymol, and carvacrol in a *p*-menthane skeleton (see Fig. 2) [18].

Table 1. Volatile constituents of *Pectis elongata* essential oils from the Amazon

Constituents (%)	RI_{CAL}	RI_{LIT}	PeSTM	PeBEL	PeBUJ	PeFEG
α-Pinene	934	932[a]	0.1			
Camphene	949	946[a]	0.1			
Sabinene	976	969[a]		1.8	0.6	
β-Pinene	978	974[a]	0.1			
6-methyl-5-Hepten-2-one	986	981[a]	1.0			
Myrcene	991	988[a]		0.2	0.1	
Octanal	1003	998[a]	0.1			
Decane	1005	1000[a]	0.1			
Limonene	1028	1024[a]	1.5	43.7	33.7	0.3
(*E*)-β-Ocimene	1046	1044[a]	0.2			
cis-Linalool oxide (furanoid)	1072	1067[a]				0.2
trans-Linalool oxide (furanoid)	1088	1084[a]				0.1
Linalool	1100	1095[a]	0.4			
exo-Isocitral	1144	1140[a]	0.3			
Citronellal	1152	1148[a]	0.2			
(*Z*)-Isocitral	1164	1160[a]	1.9			
Terpinen-4-ol	1177	1174[a]		0.1		
(*E*)-Isocitral	1182	1177[a]	2.7			
α-Terpineol	1191	1186[a]	0.1			
Nerol	1228	1227[a]	0.4			
Carvone	1242	1239[a]				0.3
Neral	1243	1235[a]	39.1			
Geraniol	1255	1249[a]	0.3			
Geranial	1270	1264[a]	47.7			
Perilla aldehyde	1272	1269[a]		51.7	64.6	81.8
Tridecene	1292	1290[a]	1.3		0.7	
Perilla alcohol	1294	1294[a]		1.5	0.1	5.6
β-Elemene	1393	1289[a]	0.2			
α-Humulene	1455	1452[a]	0.6	0.1		
Perilla acid	1500	1500[b]				4.0
(*E*,*E*)-α-Farnesene	1509	1505[a]	0.1			
Humulene epoxide II	1610	1608[a]	0.1			
Monoterpene hydrocarbons			2.0	45.7	34.4	0.3
Oxygenated monoterpenes			93.1	53.3	64.7	92.0
Sesquiterpene hydrocarbons			0.9	0.1		
Oxygenated sesquiterpenes			0.1			
Other			2.5		0.7	
Total (%)			98.6	99.1	99.8	92.3

RI_{CAL} = Calculated Retention Index (Rtx-5ms column). RI_{LIT} = Literature Retention Index. [a]Adams 2007 [13]. [b]Nist 2011 [14]. PeSTM = P. elongata current oil. PEBEL = P. elongata from Belém (Pará state) [17]. PeBUJ = P. elongata oil from Bujaru (Pará state) [17]. PeFEG = P. elongata oil from Ferreira Gomes (Amapá state) [17]

Concerning the oil composition of the current sample of *Pectis elongata* (PeSTM), collected in Santarém, Pará state, and the composition of oils of the other three *P. elongata* samples from Belém (PeBEL) [17] and Bujaru (PEBUJ) [17], Pará state, and Ferreira Gomes (PeFEG) [17], Amapá state, it is assumed that there are at least two chemical variations for this species. They are the chemotypes neral plus geranial (PeSTM oil) and perilla aldehyde plus limonene (PeBEL, PeBUJ, and PeFEG oils). When adapting to a specific environment, plants can generate varieties with different chemical characteristics but maintaining their morphology. Chemotypes are identified in plants that present differentiation in their secondary metabolism, with an evident structural variation. The analysis of these plants' chemical composition must be considered as it directly affects their biological properties. The active principles of plants differ in the composition of their chemotypes. The compositional variation observed for these different oils of *P. elongata* can be attributed to the influence of environmental conditions at

collection areas, as soil and climate, resulting in the appearance of different chemical types for the same species [20].

Three samples of *Pectis elongata* from Martinique, West Indies, have produced essential oils rich in citral (39% to 67%), composed by the monoterpene isomers, neral (15% to 27%), and geranial (24% to 40%), and which have presented significant bacteriostatic and fungistatic properties [21]. Also, in the oil of *Pectis floribunda* A. Rich. [syn. *Pectis elongata* Kunth] [11] existing in Cuba, the primary constituents were perilla aldehyde (44.5%), limonene (9.7%), *cis*-limonene oxide (8.4%), and *trans*-limonene oxide (8.2%) [22]. The first publication with the essential oil of *Pectis elongata*, rich in citral (60.0%), was from a sample collected in Puerto Rico [23].

Riparian people in the Brazilian Amazon have used a root tea of *P. elongata* to treat air sickness, which affects children and manifests with fever, fluctuations in consciousness, groans, disconnected speech, headache and neck, tremors, convulsions, salivation, and vomiting. These symptoms can be associated with tetanus, measles, and hepatitis [24]. Inhabitants of Jaú National Park, Amazonas state, Brazil, mention *P. elongata* leaf tea in genitourinary disorders. When associated with the fat of *Tapirus terrestris* L. (Tapiridae, tapir), it is also used to facilitate childbirth, among other uses [25]. The infusion of the entire plant of *P. elongata* is indicated for the treatment of hypotension in Northeast Brazil [26]. Also, the infusion of an ecotype of *P. elongata* existing in French Guyane has been used to prepare teas and spices by the Creóle population, in replacement of citronella [*Cymbopogon citratus* (D.C.) Stapf] [27].

Essential oils from other *Pectis* species have been previously reported, the oil of *Pectis texana* Cory [syn. *P. angustifolia* var. *fastigiata* (A. Gray) D.J. Keil] from the USA, showed 48% of thymol [28]. The oil of *Pectis papposa* Harv. & A. Gray, known as “cinchweed” in California and Arizona, USA, was described as rich in cumin aldehyde (47%), β-pinene (27%), and carvone (12%) [29]. In the oil of *Pectis prostata* Cav. from Cuba has predominated perilla aldehyde (70.7%) and limonene (16.2%) [30]. The oils of *Pectis apodocephala* Baker and *Pectis oligocephala* (Gardner) Sch. Bip., from Sobral, Ceará state, Brazil, were analyzed: The *P. apodocephala* oil was characterized by a significant percentage of

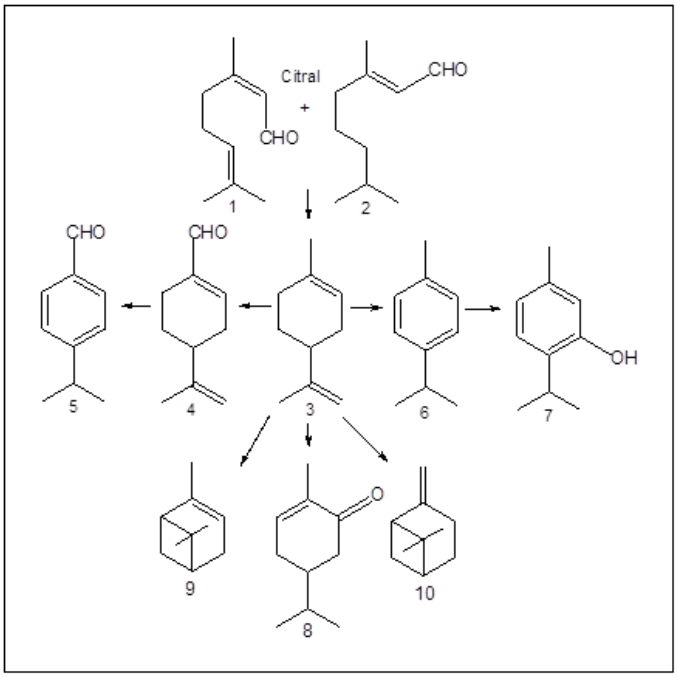

Fig. 2. Primary constituents found in *Pectis* spp essential oils

Neral (1), geranial (2), limonene (3), perilla aldehyde (4), cumin aldehyde (5), p-cymene (6), thymol (7), carvone (8), α-pinene (9), and β-pinene (10). Probable interconversion involving isomerization, cyclization, hydroxylation, and aromatization biosynthetic reactions (Dewick 2002) [19]

78% of citral (neral 34% and geranial 44%) followed by α-pinene (11%), whereas the *P. oligocephala* oil showed *p*-cymene (71%) and thymol (24%) as its major constituents [31]. Another oil from *P. apodocephala* and the oil of *P. brevipedunculata* Sch. Bip., which were sampled in the states of Ceará and Rio de Janeiro, Brazil, respectively, also presented a large amount of citral: the oil of *P. apodocephala* from Ceará displayed 53% of citral (neral 28%, geranial 25%) and the *P. brevipedunculata* oil from Rio de Janeiro exhibited 82% of citral (neral 36%, geranial 46%) [32-33]. The oil of *Pectis odorata* Griseb., sampled in Córdoba, Argentina, showed limonene (50%) and citral (50%, neral 27%, and geranial 23%) as its primary constituents [34].

The uses and properties of other *Pectis* species have been described. *Pectis papposa* has been suggested as a potential source of commercial food, as beverage flavoring, and to treat stomachache, which action is attributed to the significant content of cumin aldehyde in its essential oil [29,35]. The tea of *Pectis apodocephala*, with occurrence in Ceará, Brazil, has been described as a stomachic, tonic, and sedative agent, most likely due to its expressive content of citral and limonene [31,32]. Besides, the citral rich oil of *Pectis apodocephala* and the *p*-cymene and thymol rich oil of *P. oligocephala* Sch. Bip. have exhibited significant nematicidal and larvicidal activity against *Meloidogyne incognita* and *Aedes aegypti*, respectively [31,36]. The oil of *Pectis brevipedunculata* collected in Rio de Janeiro, Brazil, has exhibited a significant vasodilator activity in the rats' aorta [37]. In the Ethnopharmacopea of Professor Francisco José de Abreu Matos, a famous researcher of medicinal plants from Northeastern Brazil, the tea of *Pectis brevipedunculata* is mentioned for therapeutic use in the treatment of stomatitis [9]. The leaf infusion of *P. brevipedunculata* has been used in the treatment of childhood acute respiratory infection at Barbalha city, Ceará, Brazil [38].

The tea of *Pectis odorata* Griseb. is used against intestinal spasm by the Izoceño-Guaraní Indians of Chaco Seco, Bolivia, to treat the digestive disorder similar ailments in the Argentinean popular medicine, as well as anticatarrhal by the Pilagá Indians of Central Chaco of Argentina [3-5,8,39]. The oil of *Pectis odorata* existing in Córdoba, Argentina, has inhibited Gram-positive bacteria's action and exhibited cytotoxic activity against herpes and dengue viruses [34,40]. The teas of *Pectis oligocephala*, *P. oligophylla* Baker, and *P. linifolia* L. var. *linifolia* are popularly used in Northeast Brazil to treat colds, flu, gastric disorders, and hypotension [26,41]. The tea of *Pectis haenkeana* (DC.) Sch. Bip. has been used to treat gastrointestinal disorders in Mexico [6]. Infusion of *Pectis jangadensis* S. Moore, known as "erva-de- carregador" in the Pantanal region, Matogrosso, Brazil, is used as a blood cleanser and to treat diabetes [42]. Leaves and flowers of *Pectis multiseta* var. *ambigua* (Fernald) D.J. Keil are used to compose a recreational tea (healthy and medicinal) with an antipyretic effect in the Baja California Sur region, Mexico [7]. Extracts of *Pectis* species exhibited a significant light-activated biocidal action due to the presence of phototoxins, which can inhibit organisms like viruses, phytopathogenic bacteria and fungi, nematodes, and herbivorous insects [43]. The hydroalcoholic extract of *Pectis apodocephala* showed anticancer activity against Walker's tumor [44].

Citral (neral plus geranial) is found in other aromatic plants with the same scent as lemon. It is commonly used as a bacteriostatic and fungistatic agent, a citric enhancer, house odorant, and insect repellant [45,46]. Also, citral-containing essential oils, such as lemongrass oil, exhibit a broad spectrum of fungitoxicity by inhibiting several fungal species, whose fungi toxic potency remains for long food storage time [47,48].

4. CONCLUSION

This work highlighted the essential oils of *Pectis elongata*, represented by a mixture of the isomeric oxygenated monoterpenes neral and geranial, and perilla aldehyde and limonene, comprising two different chemotypes. *Pectis* spp have pleasant citrus, cumin, and oregano-like scents due to monoterpenes compounds in their volatile compositions, as citral (neral and geranial), limonene, perillaldehyde, cumin aldehyde, carvone, p-cymene, and thymol. There is a particular interest in the world's citral-rich essential oils, such as *Pectis elongata* oil and other similar *Pectis* oils, given their human health and food preservation application.

CONSENT

It is not applicable.

ETHICAL APPROVAL

It is not applicable.

ACKNOWLEDGEMENTS

The authors are grateful to CNPq and CAPES, agencies of scientific research of the Brazilian Government, and FAPESPA, the agency for the scientific investigation of the Government of the State of Pará, for their financial support.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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