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Apr. 2010, Volume 4, No.4 (Serial No.29)
Journal of Chemistry and Chemical Engineering, ISSN 1934-7375, USA

Chemical Composition of Bergamot (Citrus Bergamia Risso) Essential Oil Obtained by Hydrodistillation

Bouzouita Nabiha[1], El Omri Abdelfatteh[1], Kachouri Faten[1], Casabianca Hervé[2] and Chaabouni Mohamed Moncef[1]

1. Higher Institute of Food Industries of Tunis, 58, Avenue Alain Savary, Tunis 1003, Tunisia

2. National Centre of the Scientific Research, Echangeur de solaize, Vernaison B .P. 22-69390, France

Received: October 9, 2009 / Accepted: October 27, 2009 / Published: April 30, 2010.

Abstract: Peel essential oil of bergamot (*Citrus bergamia* Risso) growing in Tunisia was separated by hydrodistillation and obtained in a yield of 9.7%. The oil composition was investigated using GC and GC-MS with two columns HP-1 and HP-Innowax. Fifteen compounds accounting for 98.52% of the oil were identified. The oil was characterized by high content of limonene (59.21%), linalool (9.51%) and linalyl acetate (16.83%).

Key words: *Citrus bergamia* Risso, bergamot peel oil, essential oil composition, limonene, linalyl acetate.

1. Introduction

Citrus peel oils are widely used in the perfume and cosmetic industries. Among them, bergamot (*Citrus bergamia* Risso) peel oil is the most valuable essential oil due to its unique fragrance and freshness. The essence composed of a volatile part and non-volatile fraction find application in the cosmetic, pharmaceutical and food industries [1-3]. In the volatile fraction, the oxygenated compounds are included in a superior amount to that contained in other citrus essential oils extracted from peels. This oxygenated terpene fraction provides much of the characteristic flavor of bergamot oil and its high amount makes this citrus essential oil unique by its fragrance and aroma [4]. Many studies concerning the chemical composition of the peel of different varieties of citrus have been reviewed [5-9]. The bergamot essential oil produces in Reggio Calabria, Italy, has been ranked as the highest quality at the international trading market [10]. In 1994, Mondello et al. [11] studied the chemical composition of bergamot essential oil. They detected more than 100 volatile compounds in Italian bergamot oil, of which linalyl acetate and linalool were predominant in addition to limonene. In 1995, Giacomo and Mincione [12] reviewed this chemical composition.

This study aims to investigate the oil composition of bergamot peel obtained by hydrodistillation and to compare it with composition of bergamot essential oil provided by the Bergamot Consortium of Reggio Clabria, Italy [10].

2. Experiment

Bergamot fruit was collected in November 2004 from Tunisia. Grated peel (50 g) were mixed with 200 ml H_2O and subjected to hydrodistillation using Dean-Stark apparatus (until there was no significant increased in the volume of oil collection) to give the following yield (w/w): 9.7%. The oil was dried over anhydrous sodium sulfate and stored under N_2 at 4 □. The determination of retention data and the area percentage of the identified constituents were carried out on a two GC-FID systems.

Corresponding author: Chaabouni Mohamed Moncef, professor, research field: Valorization of the natural compounds of origin vegetable. E-mail: chaabouni.medmoncef@iresa.agrinet.tn.

(1) An Agilent 5890 system equipped with HP-1 (ref 1909 1 Z-115) column (50 × 320 µm, 0.5 µm film thickness). GC oven temperature was kept at 80 °C for 8 min and programmed to 220 °C at a rate of 2 °C /min.

(2) An Agilent 6890 system equipped with HP-Innowax (ref 19091 N-216) column (60 m x 320 µm i.d., 0.5 µm film thickness). GC oven temperature was kept at 60 °C and programmed to 245 °C at a rate of 2 °C /min, then constant at 250 °C for 20 min.

The split ratio was adjusted to 1/100. The injector temperature was 250 °C. The FID detector was kept at 250 °C. The carrier gas was helium (1.3 ml/min).

GC-MS analysis was carried out using the first system, Agilent 5890 equipped with HP-1 column. The mass spectra was recorded in the electron impact mode at 70 eV using the aforementioned chromatographic conditions. Individual components of bergamot peel essential oil were identified by their retention indices compared with literature values [13, 14] and their mass spectra were interpreted on the basis of the WILEY 275 L computer library.

3. Results and Discussion

The compounds identified in the Tunisian *Citrus bergamia* peel oil are listed in Table 1. The forty six identified components constituted 98.52% of the total oil. GC and GC–MS analysis showed that the oil consisted of three main groups of constituents named monoterpenes hydrocarbons, oxygenated monoterpenes and sesquiterpenes. Bergamot peel oil studied here contained 66.37% of monoterpenes hydrocarbons with limonene as the major component (59.21%). Furthermore, the oil contained 31% oxygenated monoterpenes, the main components being linalyl acetate (16.83%) and linalool (9.51%). The oil had 1.15% of sesquiterpenes. The main sesquiterpenes were *β*-Bisabolene (0.47%) and Trans *α*-Bergamotene

Table 1 Chemical composition of peel Citrus bergamia essential oil from Tunisia.

Compound	Percentage	Kovats Indices	
		HP-1	HP-Innowax
α-Thujene	0.01	924	1028
α-Pinene	0.48	932	1028
Camphene	0.02	945	1074
Sabinene	0.73	966	1126
β-Pinene	4.38	973	1118
Octanal	0.02	979	1273
Myrcene	1.23	981	1163
α-Phellandrene	0.07	993	1163
p-Cymene	0.03	1014	1271
Limonene	59.21	1029	1214
β- Phellandrene	0.14	1029	1219
(Z)-β-Ocimene	0.01	1033	1234
(E)-β-Ocimene	0.05	1037	1251
γ-Terpinene	0.01	1051	1251
(E)-Thujan-4-ol	0.01	1053	1463
(Z)-Linalool Oxide	0.02	1053	1451
(E)-Linalool Oxide	0.03	1072	1463
Terpinène	0.01	1080	1273
Linalool	9.51	1087	1542
(Z)-Limonene Oxide	0.18	1117	1451
(E)-Limonene Oxide	0.06	1124	1463
Citronellal	0.02	1131	1470
Terpinen-4-ol	0.05	1162	1601
α-Terpineol	1.09	1173	1691
Decanal	0.02	1184	1475
Octyl acetate	0.06	1192	1475
Nerol	0.28	1210	1788
Neral	0.28	1214	1681
Geraniol	0.62	1236	1833
Linalyl acetate	16.83	1244	1559
Geranial	0.27	1256	1730
Bornyl acetate	0.01	1270	1589
Terpinen-4-yl acetate	0.33	1287	-
Linalyl propionate	0.08	1331	1606
Citronnellyl acetate	0.18	1333	1681
Neryl acetate	0.4	1342	1721
Geranyl acetate	0.64	1360	1751
β-Bourbonene	0.01	1386	-
Decyl acetate	0.01	1392	-
(Z)-α-Bergamotene	0.02	1411	1574
β-Caryophyllene	0.1	1419	1606
(E)-α-Bergamotene	0.32	1433	1589
(E)-β-Farnesene	0.03	1447	1663
α-Humulene	0.01	1452	1677
γ-Muurolene	0.11	1471	1695
Germacrene D	0.02	1480	1720
Bicyclogermacrene	0.02	1493	1751
β-Bisabolene	0.47	1501	1728
Caryophyllene oxide	0.02	1571	1990
α-Bisabolol	0.01	1669	-
Total (%)	98.52		

(0.32%). The results obtained were compared with those reported in the literature (bergamot oil produced in Reggio Calabria in Italy). The proportion of limonene in bergamot peel essential oil from Tunisia is high (59.21%) compared with that in essential oil produced in Reggio Calabria (37.2%). It is know that the ratio of linalool to linalyl acetate "essence degree" is one of the quality indices of bergamot essential oil and affects the aroma of the essence of bergamot. The value of this ratio is 0.6 in the present work. The mean ratio of bergamot oil produced in Reggio Calabria has been reported to be approximately 0.3 [10].

According to the existing literature the oil of bergamot peel obtained by cold-pressing consists of limonene, linalool, linalyl acetate and non volatiles compounds such as Bergamottin, Bergapten and Citropten [1, 9] which may be deleterious. Pervaporation is reported as an alternative process to the traditional techniques (steam distillation and solvent extraction) used nowadays for extraction of aroma compounds from natural matrixes [1]. The extraction of essential oil from bergamot fruit with enzymatic pre-treatment has been studied; the enzymes have the function to degrade cell membrane and wall allowing the almost total release of aroma compounds [15]. Supercritical fluid chromatography was used in order to investigate the possibility of detoxification of this essential oil [9]. However, this purification may be detrimental to the flavor of the oil, since the processed oil will have a lower content of the oxygenated terpenes which are responsible for the olfactory characteristics [9].

4. Conclusions

The results of the oil composition obtained by hydrodistillation showed a high proportion of limonene (59.21%) and a high ratio of linalool to linalyl acetate (0.6). This oil presents a good essence degree which is better than thus of Calabria essential oil obtained by expression.

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