Phytochemistry, Vol. 26, No. 11, pp. 3005–3010, 1987.
Printed in Great Britain.

0031-9422/87 $3.00+0.00

INVESTIGATION OF THE ESSENTIAL OIL OF VIETNAMESE GINGER

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(*Revised received* 15 *March* 1987)

Key Word Index—*Zingiber officinale*; Zingiberaceae; Vietnamese ginger; essential oil; terpenes; decontamination.

Abstract—The composition of the hydrodistilled essential oil of dried Vietnamese ginger was determined by means of GC, GC-MS and ^{13}C NMR. The oil yield was 2.7% and it consisted of 28% monoterpene hydrocarbons, 37% oxygenated monoterpenes, 25% sesquiterpene hydrocarbons, 8% oxygenated sesquiterpenes and 2% non-terpenoid compounds. The composition was most similar to that of a variety of fresh Sri Lankan ginger. The main component was geranial (16%) which gave the ginger, together with neral, a lemony character. Other compounds which are supposed to be of importance for the characteristic odour of ginger were all present. Furfural, 2,6-dimethylhept-5-enal, dihydroperillene, *p*-cymen-8-ol, allo-aromadendrene, γ-muurolene, lauric acid, methylisoeugenol, γ-eudesmol, farnesal and xanthorrhizol have not been identified before in other investigations of ginger oil. The time of distillation influenced the yield, composition and odour of the oil. Decontamination of the dried ginger by means of gamma-irradiation did not influence the yield or the composition of the oil.

INTRODUCTION

Ginger (the root of *Zingiber officinale*) has been used as a spice for at least 25 centuries. It is valued both for its aromatic, volatile constituents and its spicy, pungent constituents. The latter principles are non-volatile phenolics with various sidechains, the so called gingerols, shogaols and paradols. The volatile constituents give ginger its characteristic pleasant smell, which is variously described as 'sweet, warm, rooty spicy, delicate', 'warm, green rooty spicy, characteristic citrus-like aroma' or 'harsh, warm, rooty spicy, distinctly camphoraceous' [1]. Depending on the origin (*vide infra*) a more or less lemony or camphoraceous note can be present. Ginger is of considerable economic interest and it is currently the tenth most important spice. It is used in many different foods and drinks. The oil also finds limited use in the cosmetic, pharmaceutical and perfume industry. Ginger exhibits some biological activities; it is used as a carminative and a stimulant to the gastro-intestinal tract. According to a recent study powdered ginger may be a more effective antiemetic in cases of motion sickness than the antihistaminic dimenhydrinate (Dramamine^{R}) [2].

World production of ginger is an estimated 100 000 tons on a dry basis. Most of this is for use inside the land of cultivation. Some 25 000 tons are exported which represents a value of roughly 8 [3] to 25 million dollars [1]. Ginger is cultivated in many tropical and subtropical countries. Major exporting countries are India (over 15 000 tonnes alone), Australia, Nigeria, Fiji, Taiwan and China. The highly esteemed and thus expensive Jamaican ginger is available in only limited supply.

Several reviews have appeared on ginger discussing various aspects such as history, use, cultivation, diseases, drying, production and chemical composition of the essential oil and oleoresin [1, 4–9]. Many of the above cited facts have been taken from these reviews.

Dried ginger consists of *ca* 40–60% starch, 10% proteins, 10% fats, 5% fibres, 6% inorganic material, 10% residual moisture and 1–4% essential oil. Many investigations have been carried out in the last 40 years on the composition of the essential oil [1, 10–30].

The chemical investigations carried out prior to 1953 have been adequately summarized by Guenther [31]. The most important investigations since then are summarized in Table 1. There are substantial differences between the oils of different origins. Even oils from the same region can differ significantly because usually several varieties are cultivated. Almost certainly the method of isolation and analysis plays a role as well. The identification methods become more refined each year, which is nicely illustrated by the fact that Lawrence in his investigation in 1983 [25] identified more compounds in ginger oil than in all the earlier studies combined. All together, more than 200 different volatiles have been identified in ginger oil. It is not characterised by one particular compound but rather by a mixture of various terpenes as well as some other non-terpenoid compounds.

A few investigations have been carried out on the identity of the volatiles responsible for the characteristic ginger aroma. According to Bednarczyk and Kramer [19, 21] α-terpineol, neral, geranial, β-sesquiphellandrene, *ar*-curcumene, nerolidol and cis-β-sesquiphellandrol accounted for 85% of the flavour response of a test panel. A mixture of these compounds in the correct proportion could pass as a suitable imitation of ginger. A high content of neral and geranial ('citral') imparts a lemony character to ginger. Govindarajan has made a number of rather critical remarks on the studies of Bednarczyk and Kramer [7]. His own work showed that the hydrocarbons

Table 1. Essential oil composition of ginger

Country	India	Australia	Various*	Japan
Variety or province	Kerala	Queensland	—	3 var
Fresh/Dried	fresh?	fresh	?	fr/dr
Reference	[14]	[16]	[16]	[23]
Monoterpene hydrocarbons	4	?	?	0.2–5
Oxygenated monoterpenes	8	10–35	1–3	67–75
Sesquiterpene hydrocarbons	66	45–70	55–85	10–22
Oxygenated sesquiterpenes	17	?	?	?
Non-terpenoid compounds	1	?	?	?
Individual compounds				
Camphene	1	nd	nd	0.1–2
α-Terpineol	nd	nd	nd	0.5–2
Neral	1	1–10	tr–1	8–27
Geranial	1	3–20	tr–3	13–36
Bornyl acetate	0.1	nd	nd	nd
β-Sesquiphellandrene	nd	7–11	8–12	nd
α/β-Zingiberene	36	20–28	25–30	2–14
ar-Curcumene	18	6–10	8–19	1–5
E/*Z*-Nerolidol	nd	nd	nd	nd
β-Eudesmol	nd	nd	nd	nd
E/*Z*-β-Sesquiphellandrol	nd	nd	nd	nd

*Jamaica, Nigeria, Sierra Leone, China, Cochin.

†Oven dried (60°) samples of these varieties contained on the average 50–75% less monoterpenes.

Used abbreviations: tr = trace; ? = not given; nd = not determined or not detected.

possessed no significant aroma, except for a faintly green and rubbery odour [7], but nonanal and borneol might contribute to the ginger aroma [7]. According to work of Christoph and Drawert the α-terpineol content is probably of little importance but minor ginger components such as myrcene, hexanal and linalool might be important for the aroma [32, 33]. According to MacLeod and Pieris, neral, geranial, bornyl acetete, β-zingiberene, β-eudesmol and *trans*-β-sesquiphellandrol are important for the aroma of ginger [26]. Salzer reported that citronellyl acetate together with geranial and neral are important constituents which impart freshness to the ginger oil [34]. The present authors consider it unlikely that the typical aroma of ginger will ever be completely unravelled due to the enormous complexity of the oil (*vide infra*), the many problems connected with the subjective interpretation of the odour of the individual components and the existence of many varieties.

In the following we report on the composition of the hydrodistilled oil of Vietnamese ginger which has not been investigated before. Beside this we investigated the influence of the distillation time on the yield and composition of the oil and the influence of gamma-ray treatment on the yield and the quantitative composition of the oil. Decontamination with gamma-rays may be a useful method to eliminate the microbiological contamination of dried ginger.

RESULTS AND DISCUSSION

Sundried ginger was hydro-distilled according to the method of the European Pharmacopoeia. After six hr the yield was 2.1% and after 12 hr 2.7%. The average yield for other ginger varieties is between 1 and 3%. The residual moisture content was determined as 8.7%. The oil was first investigated by means of capillary GC. This showed the oil to be a highly complex mixture and *ca* 300 separated peaks could be discerned with the column used ($n_{\text{eff}} = 55\,000$). Subsequently the oil was investigated by GC-MS. The 77 most prominent components for which a mass spectrum was recorded are numbered in Fig. 1. The individual compounds were identified by their mass spectra, as far as possible by a GC comparison with a reference sample and in certain cases by ^{13}C NMR. GC-MS analysis also showed that several sharp peaks in the chromatogram consisted of two components eluting at nearly the same time. The results are given in Table 2.

Furfural, 2,6-dimethylhept-5-enal, dihydro-perillene, *p*-cymen-8-ol, allo-aromadendrene, γ-muurolene, lauric acid, methyl isoeugenol, γ-eudesmol, farnesal and xanthorrhizol have not been detected in any of the previous studies of ginger. A comparison of the composition of Vietnamese ginger oil with those from other origins makes it clear that Vietnamese oil is very similar to that of fresh Sri Lankan ginger of the 'Chinese' variety (Table 1). The only significant difference is the presence of a large amount of bornyl acetate and β-bisabolene is the Sri Lankan oil and the presence of a large amount of α-zingiberene and β-sesquiphellandrene in the Vietnamese oil. All the other investigated oils are considerably more different.

One small experiment was carried out to see whether the time of distillation influenced the composition of the oil. According to Govindarajan little information is available on the optimum time of distillation and differences in the quality of oil related to varying periods of distillation. Distillation times varying from a few hr to as much as 20–30 hr have been mentioned in the literature [1]. As expected this influence was quite large (Table 3). In the first six hr the percentage of oxygenated monoterpenes samples of various geographic origins (in %)

Fiji	India	Sri Lanka	Sri Lanka	Trinidad	India	Japan	Vietnam
—	?	'Sidda'	'Chinese'	St. Vincent	15 var	Oshoga	Hai Nung
fresh	?	fresh†	fresh§	dried	dried?	fresh	dried
[24]	[25]	[26]	[26]	[27]	[28]	[30]	—
?	20	28	31	22	?	tr	28
?	6	19	33	33	?	72	37
13–57	62	42	30	29	?	6	25
?	3	11	5	0	?	1	8
?	6	tr	tr	16	?	?	2
?	8	14	14	tr	1–6	nd	13
?	0.1	1	1	nd	0.5–2	1	1
9–26	0.5	5	10	25	7–15	8	8
18–40	1	4	8		6–12	25	16
?	tr	4	8	nd	nd	nd	0.2
?	9	0.3	0.2	nd	nd	0.7	4
?	30	1	0.4	11	17–29	3	9
?	8	14	6	10	3–10	2	4
?	1	0.6	0.7	nd	2–5	1	1
?	0.6	5	1	nd	nd	nd	1
?	0.4	1	1	nd	nd	nd	1

Table 2. Composition of the essential oil from *Z. officinale*

Peak No.	Component	Kovats index	% in oil	Methods of identification
1	Hexanal	919	0.07	GC, MS
2	Tricyclene	971	0.23	GC, MS
3	α-Pinene	986	3.9	GC, MS, NMR
4	2-Heptanol	991	0.27	GC, MS
5	2-Heptanone	1011	tr	GC, MS
6	Camphene	1016	12.6	GC, MS, NMR
7	Furfural	1036	tr	GC, MS
8	Unknown $C_{10}H_{14}$	1042	tr	GC, MS
9	Sabinene	1047	0.07	GC, MS
10	β-Pinene	1051	0.53	GC, MS
11	Myrcene	1060	1.9	GC, MS, NMR
12	α-Phellandrene	1082	0.40	GC, MS
13	α-Terpinene (tent.)	1094	0.07	GC
14	Limonene	1106	2.1	GC, MS
15	β-Phellandrene	1117	5.7	MS, NMR
16	Octanal (tent.)	1124	0.08	GC
17	6-Methylhept-5-en-2-one	1128	0.15	GC, MS
18	*p*-Cymene	1133	5.3	GC, MS
19	1,8-Cineol	1133		GC, MS, NMR
20	γ-Terpinene	1151	0.05	GC, MS
21	2,6-Dimethylhept-5-enal	1180	0.06	MS
22	Dihydroperillene (tent.)	1184	0.35	MS
23	Terpinolene	1184		GC, MS
24	2-Nonanol	1194	0.20	GC, MS
25	Perillene isomer	1197	0.08	MS
26	Linalool	1210	0.65	GC, MS
27	2-Nonanone	1210		GC, MS
28	Perillene	1213	0.19	MS
29	Rosefuran	1221	0.18	MS
30	Unknown $C_{10}H_{16}O$	1233	0.18	

Table 2. *Continued*

Peak No.	Component	Kovats index	% in oil	Methods of identification
31	Citronellal	1286	0.29	GC, MS
32	Unknown $C_{10}H_{16}O$	1293	0.14	
33	Isoborneol	1298	tr	GC, MS
34	Borneol	1313	1.8	GC, MS, NMR
35	Camphor	1321	0.12	GC, MS
36	Unknown $C_{10}H_{16}O$	1336	0.29	
37	α-Terpineol	1342	1.0	GC, MS
38	Citronellol	1359	0.30	GC, MS
39	*p*-Cymen-8-ol	1380	0.07	MS
40	Myrtenal	1390	0.06	GC, MS
41	2-Undecanol	1397	0.05	GC, MS
42	Geraniol	1404	0.69	GC, MS, NMR
43	2-Undecanone	1418	?	GC, MS
44	Neral	1418	8.1	GC, MS, NMR
45	Bornylacetate	1428	0.21	GC, MS
46	Unknown $C_{15}H_{24}$	1453	?	
47	Geranial	1457	15.9	GC, MS, NMR
48	β-Elemene	1483	0.30	MS
49	β-Caryophyllene (tent.)	1521	0.09	GC
50	Geranyl acetate	1541	0.20	GC, MS
51	*tr*-β-Farnesene	1551	0.12	MS
52	Allo-aromadendrene	1575	0.14	GC, MS
53	α-Zingiberene	1605	9.2	MS
54	α-Farnesene	1616	7.8	GC, MS
55	β-Bisabolene	1616		MS
56	*ar*-Curcumene	1616		MS
57	γ-Muurolene	1620	0.91	MS
58	Unknown $C_{15}H_{24}$	1628	0.59	
59	β-Sesquiphellandrene	1641	4.3	MS
60	Unknown $C_{15}H_{24}$	1652	0.26	
61	δ-Cadinene	1655	0.26	MS
62	Selina-3,7(11)-diene	1655		MS
63	Lauric acid	1696	0.09	GC, MS
64	E-Nerolidol	1700	0.70	GC, MS
65	Unknown $C_{15}H_{22}$	1705	0.48	
66	Elemol	1713	0.38	MS
67	Unknown $C_{15}H_{26}O$	1745	0.27	
68	Unknown $C_{15}H_{26}O$	1767	0.69	
69	Methylisoeugenol	1774	0.08	GC, MS
70	Unknown $C_{15}H_{26}O$	1795	0.62	
71	γ-Eudesmol	1819	0.23	MS
72	β-Bisabolol	1844	0.59	MS
73	β-Eudesmol	1853	0.93	GC, MS
74	Unknown $C_{15}H_{26}O$	1872	0.57	
75	*tr*-β-Sesquiphellandrol	1899	0.72	MS
76	Farnesal	1939	0.20	MS
77	Xanthorrhizol	1986	0.10	GC, MS
			95.15	

Abbreviations used: tr = trace; tent. = tentatively identified.

was much higher and the percentage of the sesquiterpene hydrocarbons much lower than in the total oil. For the oil collected from 6–12 hr it was opposite. Quantitatively the oil collected in this last period contributed less than 25% of the oil. These results can be readily explained by two effects: (i) a volatility effect favouring the distillation of the more volatile compounds, i.e. the monoterpenes and (ii) a hydrodiffusion effect favoring the distillation of the more water soluble components, i.e. the oxygenated compounds. After the initial six hr most of the oxygenated monoterpenes have been removed from the ginger. The time of distillation is thus another variable influencing the composition of ginger oil, besides such variables as climate, variety (Table 1), drying methods [26, 27] and also identification methods.

Both the pure and diluted Vietnamese ginger oil was

Table 3. Influence of time of distillation on the essential oil composition of ginger (17 major components)

Compound	Total oil collected over 12 hr	Oil collected for first 6 hr	Oil collected for last 6 hr
α-Pinene	3.9	3.5	5.7
Camphene	12.6	12.0	15.2
Myrcene	1.9	1.8	2.3
Limonene	2.1	2.1	2.3
β-Phellandrene	5.7	5.5	6.6
1,8-Cineol	5.3	9.0	1.0
Borneol	1.8	3.1	0.5
α-Terpineol	1.0	1.5	0.3
Neral	8.1	11.9	3.0
Geranial	15.9	19.4	4.0
α-Zingiberene	9.2	3.9	16.8
α-Farnesene	7.8	3.4	12.5
β-Bisabolene			
ar-Curcumene			
β-Sesquiphellandrene	4.3	1.8	7.3
γ-Muurolene	0.9	0.4	1.4
β-Eudesmol	0.9	0.6	1.2

evaluated for its olfactory properties by a six person test panel. When the pure oil was applied to a piece of filter paper it developed initially a somewhat unpleasant pungent, camphoraceous odour. After *ca* one min this unpleasant odour had almost faded and a more pleasant citrus-lemony aroma developed. Some pleasant fatty notes could be distinguished as well. Everyone liked the overall aroma of the diluted oil in the same test (diluted × 10 with pentane). When a small volume of oil was suspended in water the fluid developed the characteristic pleasant odour of ginger. Thus Vietnamese ginger may be suitable for the production of ginger ale.

Fresh ginger roots are usually heavily contaminated with various microorganisms. Upon drying this contamination is somewhat reduced but nevertheless remains high. Plate counts of over ten million colony forming units/g of dried ginger are not unusual [1]. The dried ginger investigated by us was also quite contaminated [35]. This contamination can become a health hazard when untreated dried ginger is used in the preparation of food. Aflatoxins have also been detected in dried ginger samples [36]. To reduce the microbiological load dried ginger can be fumigated with ethylene oxide. However, various problems are connected with the use of ethylene oxide and gamma irradiation is becoming more and more popular. A few investigations have been carried out to examine the influence of irradiation on the composition, taste and odour of the oil. According to Tjaberg *et al.* after irradiation with 15 KGy the spice was sterile while no significant variations in either taste or odour could be observed [37]. These studies are more or less repeated with Vietnamese ginger. A radiation dose of 10 KGy was found to be sufficient to reduce the microbial contamination to a satisfactory level [35]. The oil yield was the same as before irradiation. Also the GC pattern was within experimental error almost identical to the GC pattern before irradiation. Only the α-pinene concentration was somewhat lower than that before irradiation. Thus gamma irradiation seems to be a good alternative for ethylene oxide for the decontamination of dried ginger.

EXPERIMENTAL

Plant material. Zingiber officinale Rosc. roots were collected in September 1985 in the Hai Hung province of Vietnam. The roots were washed, cut into pieces of *ca* 3 cm and sundried. Voucher specimens are available at the centre for Natural Products Chemistry at Ha Noi.

Hydrodistillation. Samples of 25 g were hydro-distilled in the apparatus described in the European Pharmacopoeia. A 500 ml round bottom flask was used with 250 ml dist H_2O. For the quantitative determination xylene was used in the graduated section. The oil used for the GC and GC-MS experiments was distilled without organic solvents.

GC. A chromatograph equipped with a 30 m J& W DB17 (50% phenyl, 50% methyl) column, i.d. 0.3 mm was used; split ratio 1:100; carrier gas N_2; temp. prog. 60–210°, 2.5°/min; inj. temp. 250°; det. temp. 275°; FID.

GC-MS. Ionisation energy 70 eV. The same column as used as for the GC analysis. Carrier gas He.

^{13}C NMR. A 20% soln of ginger oil was prepd in deuterated benzene. From this soln a ^{13}C NMR spectrum was recorded at 75.46 MHz. The following terpenes were identified by their characteristic resonances [38] (benzene = 126 ppm); geranial (18.09, 18.18, 26.49, 40.96, 123.79, 132.87, 161.86, 190.26); neral (18.18, 33.29, 161.86, 189.75); camphene (29.66, 38.14, 42.48, 47.82, 49.02, 100.15, 166.42); α-pinene (21.72, 47.93, 117.02, 145.00); 1,8-cineol (69.98, 73.87); myrcene (18.09, 113.51, 116.33, 139.91, 146.98); limonene (21.36, 41.94, 109.38, 150.42); β-phellandrene (20.07, 20.25, 21.72, 42.87, 110.85, 144.28); α-terpineol (45.83, 72.38); *ar*-curcumene (18.09, 21.48, 145.21), borneol (14.02, 19.32, 20.79, 45.95, 50.18, 77.35); α-zingiberene? (17.23, 20.25, 121.15).

Gamma irradiation experiments. Samples of ginger were irradiated with 6 or 10 kiloGray in the pilot plant for food irradiation in Wageningen after which the ginger was hydrodistilled as before. The yield was determined and the composition of the oils was compared with the composition of oil derived from non-irradiated ginger by means of capillary GC.

Acknowledgements—We wish to thank Ir. H. Stegeman of the State Institute for Quality Control of Agricultural Products (RIKILT) Wageningen for making available to us some unpublished results of the gamma-irradiation experiments. Further we wish to thank Mr R. Bos of the department of pharmacognosy of the State University of Groningen for his help with the interpretation of some of the MS and Mr B. van Veldhuizen for recording the ^{13}C NMR spectrum.

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