ELSEVIER

Food Chemistry 85 (2004) 59–62

Food Chemistry

www.elsevier.com/locate/foodchem

Flavour quality of dehydrated lime [*Citrus aurantifolia* (Christm.) Swingle]

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Received 7 March 2003; received in revised form 2 May 2003; accepted 2 June 2003

Abstract

The hydro-distilled volatile oils of fresh and dehydrated lime [*Citrus aurantifolia* (Christm.) Swingle] fruit were subjected to GC and GC–MS. A total of 32 compounds, constituting > 98% of the volatiles of fresh lime fruit, were identified. Five sesquiterpene hydrocarbons and two sesquiterpene alcohols were found and identified for the first time in lime fruit. The volatiles from the dehydrated (~5% moisture) lime fruit on GC–MS analysis, showed marginal quantitative changes in composition of terpene hydrocarbons, alcohols and esters, but rest of the aldehydes decreased in concentrations compared to those in the fresh lime fruit.

Keywords: *Citrus aurantifolia*; Lime; Dehydration; Volatile oil; GC–MS analysis; Terpenes

1. Introduction

The genus *Citrus* includes several important fruits such as oranges, mandarins, limes, lemons and grape fruits. The rapid growth of the citrus fruit industry in the past 25 years is due to improved economic conditions in consuming nations of the world and also due to the natural distinctive flavour of citrus. The large consumption of citrus fruit is also attributed to other types of food and beverage industry (Kale & Adsule, 1995).

Lime [*Citrus aurantifolia* (Christm.) Swingle] is a highly polyembryonic species with the fruit surface smooth, greenish-yellow in colour and thin-skinned, core solid at maturity, and juice highly acidic. Lime is used for the extraction of juice, preparation of squash, concentrates, beverages and by- products, such as citric acid and pectin etc (The Wealth of India, 1986).

The fruit is mainly relished for its flavour. The major flavour components of the fresh lime fruit have been reported as limonene, α-terpineol, 4-terpineol, 1,4-cineole, 1,8-cineole, *p*-cymene, β-pinene, β-bisabolene, citral, geranial and neral (Ranganna, Govindrajan, & Ramana, 1983; Shaw, 1979). Recent works on lime, using column chromatography, GC, GC–MS and GC/FTIR, showed the presence of some more compounds, such as neryl acetate, α-bergamotene, valencene and germacrene-d (Chamblee & Clark, 1997; Chamblee, Karelitz, Radford, & Clark, 1997; Della Porta, Reverehon, Chouchi, & Barth, 1997; Dugo, Cotroneo, Bonarcorsi, & Mondello, 1998; Tincutta & Richard, 2001; Venkateswarlu & Selvaraj, 2000).

Lime is dried to prepare peel powder. In the Middle East, whole lime fruit, which is dried to a charred colour, is used in rice-based biryani/pulav-type preparations, as well as curry preparations. The powdered fruit is also sprinkled over roasted meat. Lime volatile extract also showed insecticidal properties (Ezeonu, Chidume, & Udedi, 2001). Dehydration of fruits is commonly done in different parts of the world to improve the keeping quality and to preserve the fruit for consumption during off seasons. As a result of dehydration, the water activity of fruit/product is controlled, whereby chances of microbial spoilage are minimized. Dehydrated whole lime is a unique product, consumed mainly in the Middle East. However, no reports were available on systematic drying of lime fruit, nor on the changes that occur in its flavour profile as a result of

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doi:10.1016/j.foodchem.2003.06.002

processing, though the fruit is valued mainly for its flavour.

A process was developed at the Central Food Technological Research Institute (CFTRI) for the preparation of whole dried lime (Chauhan, Rekha, Yadav, & Ramteke, 2002). Though fruit was dried to a charred colour, its flavour closely resembled that of fresh fruit. Hence, studies were carried out to determine the changes in volatile flavour components of dehydrated lime fruit.

2. Materials and methods

2.1. Plant material

Fresh lime fruits were procured locally and a voucher specimen was deposited at the Manasgangotri herbarium (MGH No. CFTRI 2A and 2B/2001), Botany Department, Mysore University, Mysore, India. The fruits were thoroughly washed, wiped with a cloth to remove surface moisture, smeared with olive oil and sugar, loaded onto trays and whole fruits were dried at the temperature of 110 °C to get black/charred coloured lime of ~5% moisture content. The dried fruits were cooled to room temperature, packed in low density poly ethylene pouches and stored at 2 °C for further use.

2.2. Isolation of volatile oil

The lime fruit samples (100 g), with fresh lime moisture content: 86.5% and dehydrated lime moisture content 4.6%, were crushed and subjected to hydro-distillation, separately, in the Clevenger type apparatus for 4 h. The oil obtained per 100 g fruit sample was dried over anhydrous sodium sulphate and stored at −18 °C prior to further analysis.

2.3. GC analysis

The GC analysis was carried out on a Fisons GC-8000 series, CE Instrument, Thermo Quest Italia, Italy, equipped with a FID detector, using a SE-30 column (10′ × 0.125″) containing 5% polydimethyl siloxane on chromosorb. The temperature was programmed from 50 °C for 2 min to 250 °C at the rate of 4 °C/min, at which temperature the column was maintained for 4 min. The injector port temperature was 250 °C, the detector temperature was 250 °C, and nitrogen was the carrier gas at a flow rate of 40 ml/min. Peak areas were computed by data processor.

2.4. GC-MS analysis

The oil was analyzed using a Shimadzu 17A-GC chromatograph, Japan, equipped with a QP-5000 (Quadrupole) mass spectrometer. The samples were diluted 20 times with acetone and 1 μl was injected. A fused silica column, SPB-1, SIGMA, USA (30 m × 0.32 mm, film thickness 0.25 μm) coated with polydimethylsiloxane, was used. Helium was the carrier gas at a rate of 1 ml/min. The injector port temperature was 250 °C, and the oven temperature was maintained at 50 °C for 2 min and then increased to 250 °C at the rate of 2 °C/min at which temperature the column was maintained for 5 min. The split ratio was 1:40 and the ionization voltage − 70 eV.

Retention indices for all the compounds were determined according to the Kovats method using *n*-alkanes as standards (Jennings & Shibamoto, 1980). The identification of the compounds was done by comparison of Kovats indices (Davies, 1990; Jennings & Shibamoto, 1980) and by matching their fragmentation pattern in mass spectra with those of the NIST library database and published mass spectra (Adams, 1989; Jennings & Shibamoto, 1980; Noever de Brauw, Bouwman, Tas, & La Vos, 1988; Strenhagen, Abrahamsson, & Mc Lafferty, 1974).

3. Results and discussion

The volatile fractions from the samples of lime fruit were isolated by hydro-distillation to obtain flavour compounds. It was found that the loss in the essential oil yield during extraction from dehydrated sample compared to the oil obtained from fresh fruit was about 30%. The yield of oil from lime fruit was 0.2 and 0.99 mℓ for 100 g fresh and dehydrated fruits, respectively (Table 1).

GC–MS analysis of fresh sample showed the presence of 32 compounds (Table 2) and constituted ~98% of the volatiles. The compounds present in the volatile fractions may be classified as hydrocarbons (75%), alcohols (18%), aldehydes (4%), and esters (1%). Limonene (37.8%) was the major component followed by β-pinene (16%) γ-terpinene (9.5%), nerolidol (7.1%) and α-terpineol (6.7%). Four aldehydes (neral, geranial, dodecanal and tetradecanal) and two esters (neryl acetate and geranyl acetate) were also found to be present. Monoterpene hydrocarbons and their oxygenated compounds constituted the major fraction (~83%),

Table 1
Yield of oil in fresh and dehydrated lime

Sl.No.	Fruit/sample	Moisture (%)	Essential oil (ml/100 g)	
			a	*b*
1	Fresh	86.5±0.5	0.20±0.006	0.20±0.006
2	Dehydrated	4.6±0.1	0.99±0.008	0.14±0.001

a = On dried fruit basis; *b* = on fresh fruit basis.

Table 2
Composition of volatile oils of fresh and dehydrated lime

Kovats indices	Compounds[b]	Quantity (μl)/100 g[a]	
		Fresh	Dehydrated
933	α-Pinene	6.8	6.6
979	β-Pinene	32.1	17.6
1041	Limonene[c]	75.5	53.5
1064	cis-Ocimene	0.7	Tr[d]
1078	γ-Terpinene	19.0	18.1
1093	Terpinolene	3.2	5.1
1102	Linalool	1.3	0.6
1107	Fenchol	0.4	1.2
1173	4-Terpineol[c]	6.8	4.7
1196	α-terpineol[c]	13.3	10.2
1227	Neral	1.8	0.3
1258	Geranial	4.1	0.6
1329	δ-elemene	0.6	1.1
1351	Neryl acetate	0.5	0.4
1370	Geranyl acetate	0.6	0.5
1380	β-Elemene	0.6	1.0
1395	Dodecanal	1.0	0.4
1405	α-Cedrene	4.5	3.4
1434	α-Bergamotene	4.7	3.5
1438	α-Humulene	0.4	0.3
1446	(Z)-β-Santalene	0.2	0.2
1452	(Z)-β-Farnesene	0.6	0.4
1461	Germacrene-d	0.7	0.2
1474	Valencene	Tr	0.1
1510	Nerolidol	14.3	6.4
1515	(E)-β-Farnesene	Tr	0.1
1520	(E)-γ-Bisabolene	0.2	0.1
1533	γ-Elemene	0.5	0.2
1584	α-Bisabolol	0.2	tr
1594	Tetradecanal	0.4	0.1
1663	β-Bisabolol	0.4	0.1
1976	Hexadecanoic acid	1.3	0.1

[a] Fresh fruit basis.

[b] Identified by KI, MS.

[c] Confirmed by co-injection also.

[d] Trace.

followed by sesquiterpenes and their oxygenated compounds (∼14%). The remaining part contained straight chain aldehydes and an acid. Hence, the volatile fraction was found to be dominated by mevalonic acid metabolites and was devoid of shikimic acid derivatives. Dugo et al. (1998) studied volatile oil composition of four lime samples from Mexico, one each from Peru and the Ivory Coast. They reported monoterpene hydrocarbons (68–72%), sesquiterpene hydrocarbons (3–6%), aldehydes (0.2–0.6%), and esters (0.1–0.3%). In the present study, alcohols are the most abundant oxygenated compounds and are present to the extent of 13–15%. Ethers and oxides combined are present to the extent of 8–14% of whole oil. Hence, overall comparison indicates that the lime fruit used in this study is one of the superior varieties with high amounts of characteristic flavour compounds of lime (aldehydes and esters). Two alcohols (β-bisabolol and nerolidol), and five sesquiterpene hydrocarbons (α-cedrene, β-(*Z*)-santalene, valencene, trans-β- farnesene and *trans*-γ-bisabolene) were reported for the first time from lime volatiles.

Similar to the fresh fruit volatiles, monoterpenes and their oxygenated compounds constituted the major fraction in the dehydrated sample, with limonene as the major component. Quantities of terpene hydrocarbons (limonene,α-pinene,β-pinene,γ-terpinene,α-cedrene, germacrene-d and *cis*-ocimene) were found to decrease compared to fresh fruit. Aldehydes (neral, geranial, tetradecanal and dodecanal) decreased, substantially during dehydration. Alcohols were also found to have decreased excepting fenchol. However, some of the hydrocarbons, such as terpinolene, δ-elemene, and β-elemene, were found to increase due to dehydration of alcohols. Esters remained almost the same. α-Terpineol, 1,4-cineole and 1,8-cineole have been shown to be derived from limonene by hydration and dehydration reactions under the acid conditions during processing, while citral is transformed to *p*-cymene and *p*-α-dimethylstyrene. γ-Terpinene, rather than citral, has been shown to be the precursor of *p*-cymene (Ranganna, Govindarajan, & Ramana, 1983; Kefford & Chandler, 1970).

In the dehydrated lime fruit sample, all volatile components were found to be present, when compared with fresh fruit, though changes were noticed in their quantities. The overall flavour quality of the dried fruit changed accordingly. This is the first report on volatiles of dehydrated whole lime fruit.

4. Conclusion

GC–MS analysis of fresh lime sample showed the presence of 32 compounds. Two alcohols and five sesquiterpene hydrocarbons were found and identified for the first time in lime fruit. High temperature drying reduced the quantities of many of the flavour compounds and overall flavour quality accordingly.

Acknowledgements

We express our gratitude to Professor Dr. G.R. Shivamurthy, Botany Department, Mysore University, India, for identifying the lime species. We thank Mr. Shivaswamy, Central Instrumentation Facilities and Services, Central Food Technological Research Institute, for assistance in carrying out the GC–MS analysis.

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