

Volatiles from herbs and spices

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11.1 Introduction

Plants contain an enormous range of isoprenoid compounds with a wide variety of structures and functions. The majority of isoprenoids are synthesized as secondary metabolites that are uniquely plant products (Bramley 1997). Isoprenoids form an integral part of the volatiles from spices and herbs. Volatile oils are chemically complex mixtures, often containing in excess of 100 individual components. Most oils have one to several major components, which impart the characteristic odour/taste, but the many minor constituents also play their part in producing the final product.

Volatile oils, which are used for culinary, pharmaceutical, and perfumery purposes, are composed of two classes of compound, terpenes and phenyl propenes. Of these, the terpenes are by far the more abundant but phenyl propenes are usually the major flavour/odour factors. The high levels of some of these compounds in turpentine oil gave rise to the alternative generic name 'terpenoid'. Terpenoids are the ingredients of perfumes, soaps, flavourings and food colourants. Terpenes constitute a major group, which contain more than 1000 monoterpenes and 3000 sesquiterpene structures (Waterman 1993).

The development of chromatographic and spectroscopic techniques has led to general understanding of structure, biosynthesis and properties of terpenoids. Terpenoids are built up of isoprene (C_5) units and the nomenclature of the main classes reflects the number of isoprenoid units present (Bramley 1997).

11.2 Classification of volatiles

11.2.1 Terpenes

Terpenes found in volatile oils can be subdivided into monoterpenes, which have a 10-carbon skeleton and sesquiterpenes, which have a 15-carbon skeleton. Diterpenes (20-carbon units) do occur in some oils (e.g. ginger). The feature that binds all these

compounds together is the presence of a 5-carbon building block, which is referred to as the isoprene unit. Table 11.1 illustrates the classes of isoprenoids found in plants. Compositional changes occur in essential oils due to the (i) effect of extrinsic conditions (ii) effect of interspecific and infrastructure differences (iii) effect of ontogeny (iv) effect of processing parameters and (v) effect of adulteration (Chikuenshu and Lawrence 1997).

Monoterpenes

In monoterpenes, it is usually possible to detect the presence of two of these isoprene units and in sesquiterpenes, three. Figure 11.1 depicts the structure of an isoprene unit. Monoterpenes can be divided into three sub groups (i) acyclic, no ring systems (ii) monocyclic, one ring and (iii) bicyclic, two rings (Fig. 11.2). Further proliferation occurs through addition (oxidation) or removal (reduction) of double bonds, and by addition of oxygen to form alcohols (-OH), ketones (-CO), aldehydes (-CHO) and esters (-OCO-).

Sesquiterpenes

Sesquiterpenes, because they possess five more carbons than the monoterpenes, have far greater potential for structural and stereo chemical diversity. Sesquiterpenes form the largest class of terpenoids and are found in plants, liverworts, mosses, fungi and algae. They commonly occur with the monoterpenoids in essential oils. They are less volatile and have less direct organoleptic properties, than monoterpenes. They are an essential part of most volatile oils, subtly influencing odour (Waterman 1993, Bramley 1997).

11.2.2 Phenylpropenes

The skeleton of phenylpropenes invariably consists of a 6-carbon aromatic ring with 3-carbon side chain attached. The side chain always contains a double bond but only

Table 11.1 Main classes of isoprenoids found in plants

Carbon atoms	Name	Parent isoprenoid	Sub-class
10	Monoterpenoids	GPP	Iridoids
15	Sesquiterpenoids	FPP	Abscissic acid, sesquiterpene lactones
20	Diterpenoids	GGPP	Gibberellins
25	Sesterpenoids	GFPP	None
30	Triterpenoids	Squalene	Phytosterols, saponins, cardenolides
40	Tetraterpenoids	Phytoene	None
740	Polyprenols, rubbers	GGPP+ (C ₅) n	None

GPP – Geranyl pyrophosphate, FPP – Farnesyl pyrophosphate, GGPP – Geranyl geranyl pyrophosphate, GFPP – Geranyl farnesyl pyrophosphate.

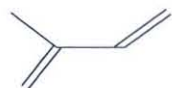
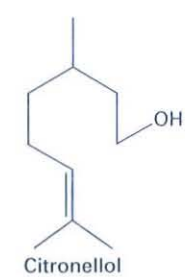
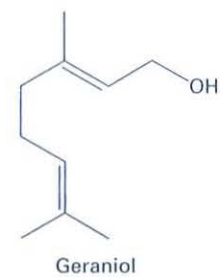
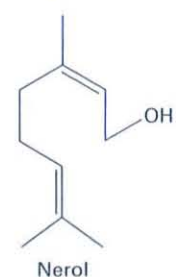
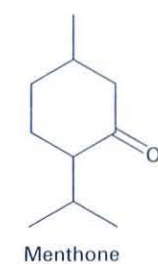
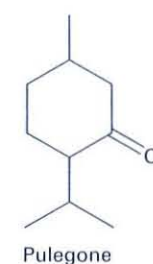
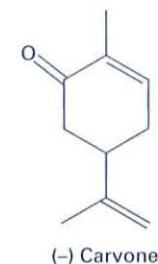
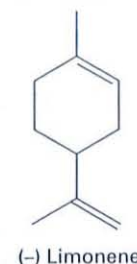


Fig. 11.1 An isoprene unit.

Acyclic monoterpenes



Monocyclic monoterpenes



Bicyclic monoterpenes

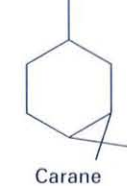
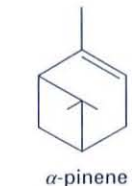


Fig. 11.2 Structures of selected monoterpenes.

occasionally an oxygen functional group (e.g. cinnamaldehyde in cinnamon oil). The aromatic ring may be substituted with up to four oxygens, which are then further modified themselves by the addition of a methylenedioxy ring, as in safrole.

11.3 Biosynthesis of the components of volatile oils

Chemicals produced by plants that are characterized by a limited distribution, and an absence of obvious value in the physiology of the producer plant, are known as secondary metabolites. The array of secondary metabolites, which of course includes volatile oils, is enormous. The terpenes constitute a major group, with more than 1000 monoterpene and perhaps 3000 sesquiterpene structures known. By contrast, the number of phenylpropenes is small, with probably less than 50 being known (Waterman 1993).

Despite the vast numbers and structural diversity of secondary metabolites, almost all arise from one of the three biosynthetic pathways, or from a combination of two or more of these pathways. These are known as the acetate, mevalonate (based on

mevalonic acid) and shikimate (based on shikimic acid) pathways. The terpenes are wholly mevalonate derived whereas the phenylpropenes originate from shikimic acid. Figure 11.3 illustrates the sequence of formation of methyl chavicol from phenylalanine by the shikimic acid pathway. Figure 11.4(a)–(d) illustrates the general biosynthetic pathway from mevalonic acid to sesquiterpenes.

11.3.1 Biosynthesis of monoterpenes and sesquiterpenes

Mevalonic acid is a chemical intermediate containing six carbons that is formed in the plant by the combination of three molecules of acetate, which have, in turn, been derived from acetyl coenzyme A. This is a universal process in all higher plants and produces compounds vital to the life processes. The biosynthesis of mono- and sesquiterpenes from mevalonic acid involves three steps: (i) conversion of mevalonic acid to isopentenyl pyrophosphate (IPP) and 3,3-dimethyl allyl pyrophosphate (DMAPP), (ii) combination of IPP and DMAPP to give geranyl pyrophosphate (GPP) and (iii) combination of GPP with IPP to give farnesyl pyrophosphate (FPP). IPP is the initial product formed from mevalonic acid and it is then converted into DMAPP by the enzyme isopentenyl pyrophosphate isomerase (Gershenzon and Croteau 1990, Waterman 1993).

One molecule of IPP and one molecule of DMAPP combine under the influence of geranyl pyrophosphate synthase to give geranyl pyrophosphate (GPP), the first recognizable monoterpene. This process is then continued by the addition of another

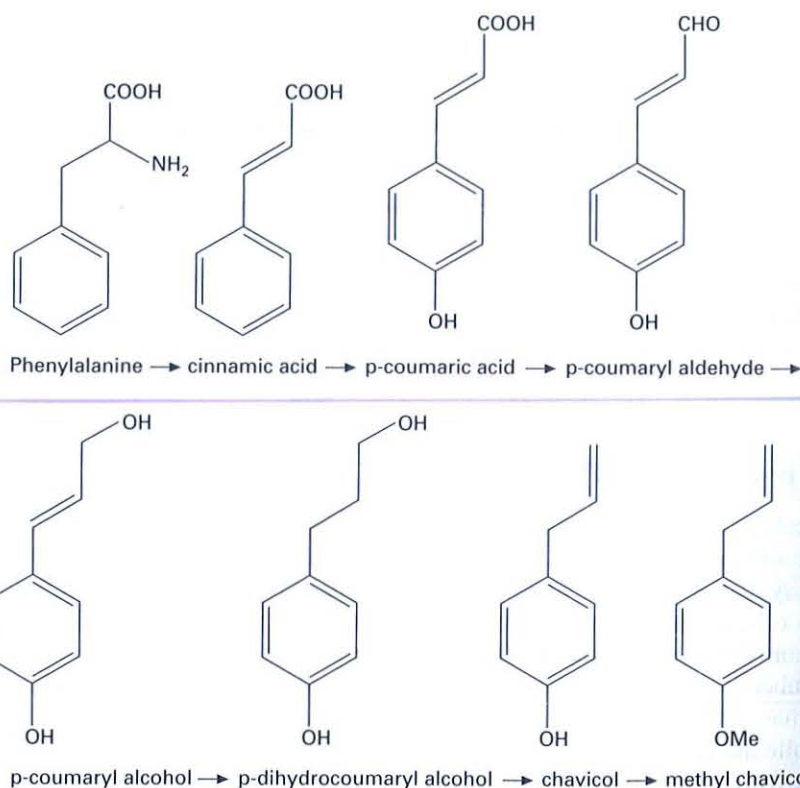


Fig. 11.3 Formation of methyl chavicol from phenyl alanine by shikimic acid pathway.

IPP to GPP through the mediation of a further synthase enzyme, resulting in the production of the first 15-carbon unit, farnesyl pyrophosphate (FPP).

11.3.2 Biosynthesis of phenylpropenes

Shikimic acid is formed from glucose in plants, and is the biogenic precursor of the amino acids L-phenylalanine, L-tyrosine and L-tryptophan. Pathways from shikimic acid generate anthranilates (e.g. in mandarin oil *Citrus reticulata*), cinnamates (e.g. in Peru balsam oil *Myroxylon pereirae*) and other phenylpropanoids, and from this point on to other metabolites such as lignans and flavononoids. In particular, phenyl propanoids (basically compounds with a 3-carbon chain attached to a benzene ring) are formed from trans or (E)-cinnamic acid via the elimination of ammonia from L-phenylalanine. Common phenylpropanoids in essential oils include methyl chavicol, methyl eugenol, eugenol, methyl cinnamate, vanillin and anethole. The shikimic acid pathway produces the amino acid phenylalanine which by the action of phenyl alanine ammonia lyase is converted to trans-cinnamic acid (Bramley 1997, Waterman 1993).

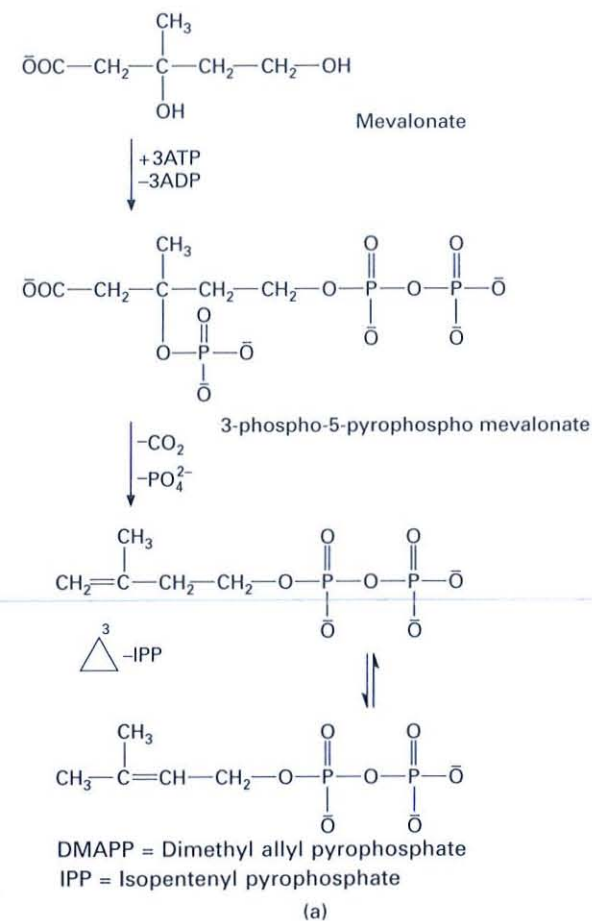


Fig. 11.4 (a) Conversion of mevalonate into activated isoprene units (Source: Nelson and Cox (2001)); (b) formation of GPP and FPP from DMAPP (Source: Nelson and Cox (2001)); (c) formation of monoterpenes from GPP (Source: Waterman (1993)); (d) formation of sesquiterpenes from EPP (Source: Waterman (1993)).

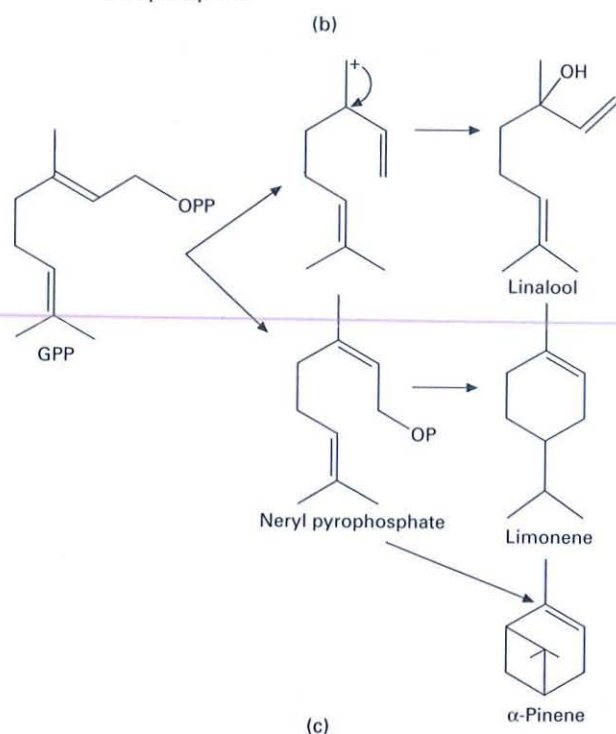
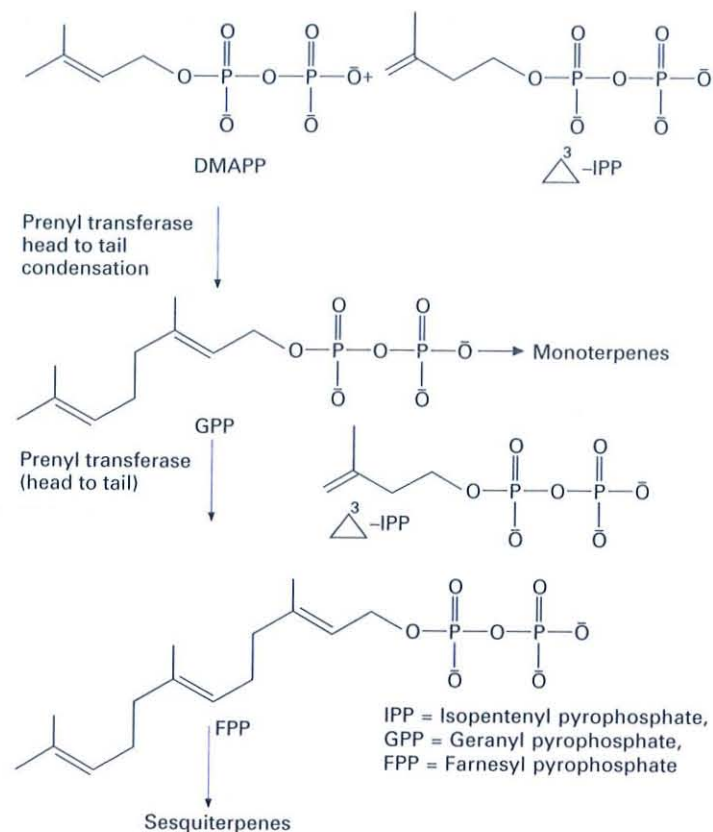


Fig. 11.4 continued

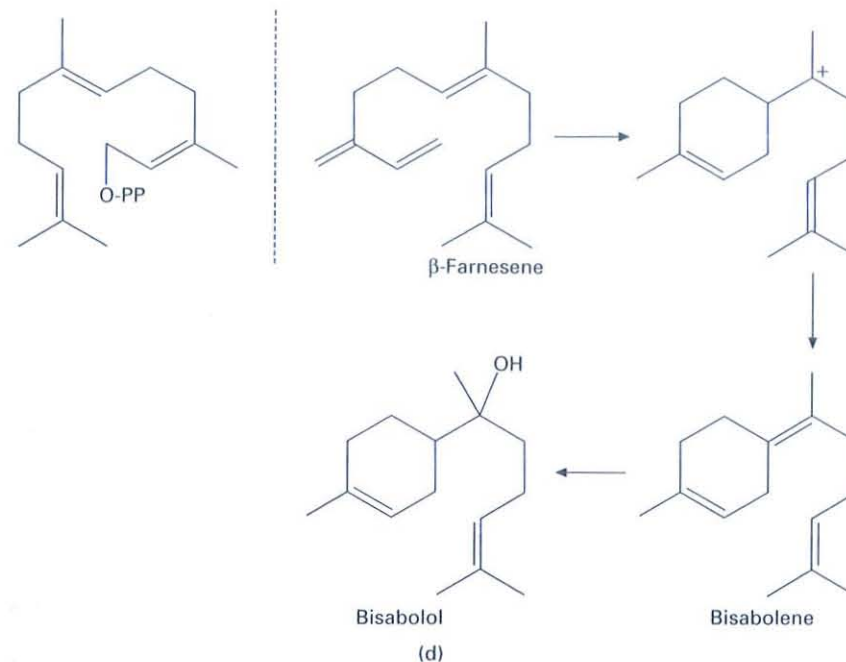


Fig. 11.4 continued

11.4 Volatiles and plant sources

The spices and herbs discussed here consist of black pepper, cardamom, ginger, turmeric, cinnamon, cassia, clove, nutmeg, cumin, coriander, fennel, fenugreek, ajowan, asafoetida, basil, mint, spearmint and rosemary. The chief chemical constituents of these spices and herbs are listed in Table 11.2.

11.4.1 Major volatiles in herbs and spices

The common volatiles found in spices and herbs are as follows:

Monoterpene hydrocarbons

Camphene, δ -3-carene, p-cymene, limonene, myrcene, cis-ocimene, α -phellandrene, β -phellandrene, α -pinene, β -pinene, sabinene, α -terpinene, γ -terpinene, terpinolene, α -thujene.

Oxygenated monoterpenes

Borneol, camphor, carvacrol, cis-carveol, trans-carveol, carvone, carventanacetone, 1,8-cineole, cryptone, p-cymen-8-ol, p-cymen-8-methylether, dihydrocarveol, dihydrocarvone, linalool, cis-menthadien-2-ol, 3,8,(9)-p-menthadien-1-ol, 1(7)-p-menthadien-6-ol, 1(7)-p-menthadien-4-ol, 1,8(9)-p-menthadien-5-ol, 1,8(9)-p-menthadien-4-ol, cis-p-2-menthen-1-ol, myrtenal, myrtenol, methyl carvacrol, trans-pinocarveol, pinocamphene, cis-sabinene hydrate, trans-sabinene hydrate, 1-terpinen-4-ol, 1-terpinen-5-ol, α -terpineol, 1,1,1,4-trimethyl cyclo-hepta-2, 4-dien-6-ol, phellandral, piperitone, citronellal, nerol, geraniol, isopinocampone, methyl citronillate, methyl geranate, α -terpinyl acetate, terpinolene epoxide and trans-limonene epoxide.

Table 11.2 Principal constituents of certain spices and herbs

Spice	Average volatile oil (%)	Total volatile oil ml/100 g (range)	Average NVEE (%)	Principal volatile constituents
Basil (Sweet)	0.4	–	3.6	Methyl chavicol, linalool, methyl cinnamate, cineole, eugenol
Cardamom	4.0	2.0–8.0	–	α -Terpinyl acetate, 2-terpineol, limonene, cineole, borneol, linalyl acetate, linalool, α -terpineol
Cassia	2.5	0.5–5.0	4.0	Cinnamic acid, benzaldehyde, methyl salicylaldehyde, cinnamic aldehyde, cinnamyl acetate
Cinnamon	0.75	0.5–2.0	5.0	Cinnamic aldehyde, eugenol, caryophyllene
Clove	16.0	12.0–20.0	7.0	Eugenol, eugenol acetate, caryophyllene
Coriander	0.3	0.0–0.1	16.0	d-linalool, d-2-pinene, dl- α -pinene, geraniol
Cumin	2.5	2.5–4.5	20.0	Cuminaldehyde, p-cymene, dihydro cuminaldehyde
Dill	3.0	2.0–4.0	17.0	Carvone, d-limonene, phellandrene
Fennel	3.0	3.0–4.0	15.0	Anethole, fenchone, d- α -pinene
Fenugreek	Trace	0.02	7.0	δ -cadinene, α -cadinol, γ -eudesmol
Ginger	2.0	1.0–3.0	5.0	D-camphene, zingiberene α - and β -phellandrene
Mace	12.0	7.0–14.0	23.0	d- α -pinene, d-camphene, myristicin, elemicin
Nutmeg	6.5	7.15	28.0	Myristicin, geraniol, d-camphene, dipentene, pinenes, safrole, p-cymene
Pepper (black)	2.5	1.0–3.0	5.5	α -pinene, β -pinene 1- α -phellandrene β -caryophyllene, limonene
Peppermint	–	0.4–1.0	–	l-menthol, l-limonene, l-menthone, α -pinene, phellandrene, d-menthone.
Spearmint	–	–	–	l-carvone, l-limonene 1-phellandrene
Turmeric	–	1.3–5.5	7.0	Zingiberene, borneol, d-sabinene, tumerone, ar-turmerone, d- α -phellandrene, cineole

Sesquiterpene hydrocarbons

β -Caryophyllene, α -cis-bergamotene, α -trans-bergamotene, β -bisabolene, δ -cadinene, γ -cadinene, calamenene, α -copaene, α -cubebene, β -cubebene, ar-curcumene, β -elemene, δ -elemene, β -farnesene, α -guaiene, α -humulene, γ -humulene, isocaryophyllene, γ -muurolene, α -santalene, α -selinene, β -selinene, ledene, sesquisabene and zingiberene (Purseglove *et al.* 1981a).

Oxygenated sesquiterpenes

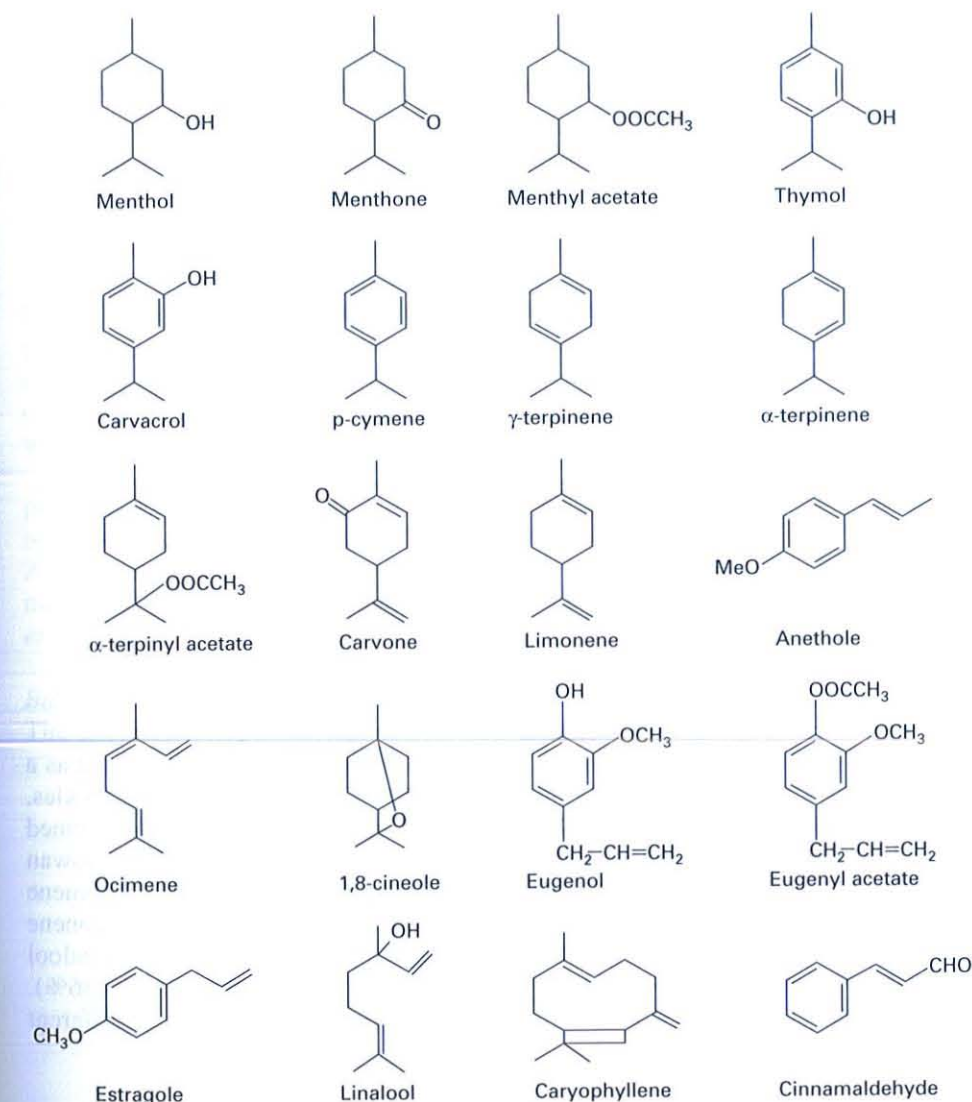
Oxygenated sesquiterpenes identified are 5, 10 (15) cadinen-4-ol, caryophylla-3-(12),7(15)-dien-4- β -ol, caryophylla-2,7(15)-dien-4- β -ol, caryophylla-2-7(15)-dien-4- β -ol, caryophyllene alcohol, caryophyllene ketone, caryophyllene oxide, epoxydihydrocaryophyllene, cis-nerolidol, 4,10,10-trimethyl-7-methylene bicyclo-(2.0)decane-4-carboxaldehyde, γ -eudesmol, elemol, cubebol, α -bisabolol, β -bisabolol, virideflorol, cubebol, epi-cubanol, turmerone, ar-turmerone and turmerol.

Miscellaneous compounds

Eugenol, methyleugenol, benzaldehyde, trans-anethole, myristicin, safrole, piperonal, m-methyl acetophenone, p-methyl acetophenone, n-butyrophenone, methyl heptanone, pinol, methyl heptanate, methyl octanoate, 2-undecanone, n-nonane, n-tridecane and aromatic acids such as benzoic acid, phenyl acetic acid, cinnamic acid, piperonic acid, butyric acid, 3-methyl butyric acid, hexanoic acid and 2-methyl pentanoic acid. The structures of the selected compounds are depicted in Fig. 11.5(a) and 5(b) (Purseglove *et al.* 1981a).

11.4.2 Volatile oil constituents

The details of volatiles from individual spices and herbs are discussed below.

**Fig. 11.5** Major volatiles from spices and herbs

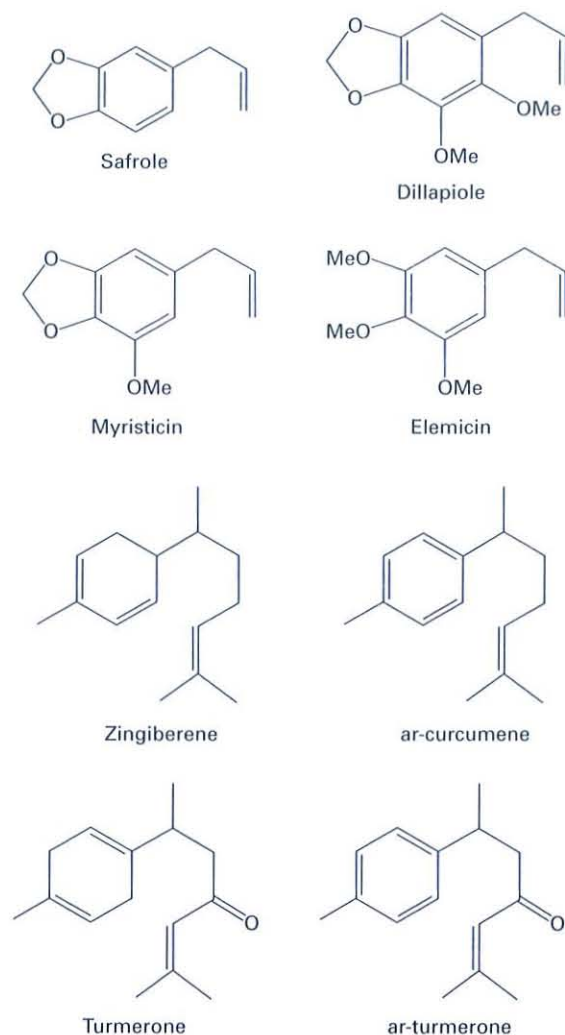


Fig. 11.5 continued

Ajowan

Ajowan or bishop's weed is cultivated for its fruits, which are commonly used as a spice and medicine. It is used for its characteristic smell and pungent taste in pickles, certain biscuits, confectionery and beverages. Nagalakshmi *et al.* (2000) determined the physicochemical characteristics of ajowan volatile oil. GC-MS profile of ajowan seed volatile oil indicated the composition as follows: α -pinene (1.48%), β -pinene (5.45%), β -myrcene (1.40%), α -terpinene (0.09%), p-cymene (19.47%), limonene (0.48), γ -terpinene (30.97%), p-cymene (0.06%), menth-2-en-1-ol (0.13%), linalool (0.07%), terpinene-4-ol (0.12%), α -terpineol (0.12%) and thymol (39.36%). They have also reported the variability in the constituents from seeds of different locations.

Asafoetida

The spice asafoetida is the dried latex (gum oleoresin) exuded from the living

underground rhizome or tap root of several species of *Ferula* (three of which grow in India), which is a perennial herb (1 to 1.5 m high). It is greyish-white when fresh, darkening with age to yellow, red and eventually brown. It is sold in blocks or pieces as a gum and more frequently as a fine yellow powder, sometimes crystalline or granulated. Studies conducted in Pakistan on fresh mature seed oils of *Ferula foetida* Regel indicated presence of α -pinene (1.69–2.36%), camphene (0.9–1.04%), myrcene (2.0–2.5%), limonene (0.60–0.72%), longifolene (1.60–5.9%), caryophyllene (3.8–5.0%), β -selinene (15.2–17.2%), eugenol (4.68–5.00%), bornyl acetate (2.25–4.5%), fenchone (1.5–2.4%), linalool (0.05–0.06%), geraniol (0.05–0.08%), isoborneol (0–0.4%), borneol (0–0.15%) and guaicol (0.57–0.9%). The oil was also found to contain a mixture of sesquiterpene alcohols (0–39.32%) and a mixture of coumarins (7.5–7.8%) (Ashraf and Bhatti 1979).

The major constituents of asafoetida are the resin (40–64%), gum (25%) and essential oil (10–17%) (Abraham *et al.* 1979). The aroma of asafoetida is attributed mainly to secondary butyl propenyl disulphide. Using MS, NMR, IR and UV spectra these were further characterized as 1-methyl propyl-(1-propenyl) disulphide (secondary butyl-(1-propenyl)-disulphide), 1-methyl thiopropyl-(1-propenyl) disulphide and 1-methyl propyl-(3-methylthio-2-propenyl) disulphide (sec.butyl-(3-methylthioallyl)-disulphide): the composition of these in asafoetida oil is 36–84%, 9–31% and 0–52%, respectively (Abraham *et al.* 1979, Lawrence 1981) (Fig. 11.6).

Pakistan sample of asafoetida contained 1-(methylthio)-propyl-(E)-1-propenyl disulphide (37.93%), 1-(methylthio)-propyl-Z-1-propenyl disulphide (18.46%), 2-butyl-(E)-1-propenyl disulphide (11.17%), dibutyl trisulphide (1.82%), isobutanol (7.65%), methyl-(E)-1-propenyl disulphide (1.69%) as major compounds (Noleau *et al.* 1991). Essential oils extracted from asafoetida gums contained more than 150 compounds of which 25 compounds, including 13 sulphur-containing compounds, were common to both leek and asafoetida (Noleau *et al.* 1991).

The oil from Iran was constituted by α -pinene (2.1%), sabinene (1.0%), β -pinene (5.0%), myrcene (1.0%), α -phellandrene (2.4%), β -phellandrene (2.5%), Z- β -ocimene (11.5%), E- β -ocimene (9.0%), 2-butyl-1-propyldisulphide (0.6%), 2-butyl-Z-1-propenyldisulphide (3.9%), 2-butyl-E-1-propenyldisulphide (58.9%), di-1-methylpropyl disulphide (0.3%) and di-1-methyl-propenyl disulphide (1.2%) (Sefidkon *et al.* 1998).

Basil

The chemical composition of volatile oils obtained from two forms of sweet basil

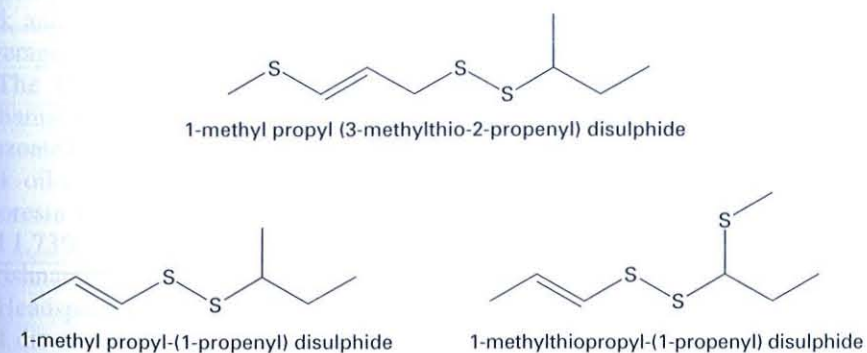


Fig. 11.6 Volatiles from asafoetida.

(purple basil and green basil) by GC-MS indicated about 35 compounds. Major compounds are eucalyptol and linalool. Estragole was not found in purple form while a low percentage was found in the green basil (Wjerdak 2001). The main constituents identified in basil condensate are linalool and methyl chavicol (Machale *et al.* 1997). Linalool and 1,8-cineole comprised more than 50% of total yield of sweet basil oil. Volatiles in fresh leaves was about 50-fold higher than those found in air dried leaves (Loughrin 2003). Sanda *et al.* (1998) described the chemical composition of *Ocimum* species growing in Togo. Linalool, estragole and α -bergamotene are the major compounds (Table 11.3).

Black pepper

Gopalakrishnan *et al.* (1993) studied four genotypes of pepper using GC-MS. The oil of these cultivars possessed α -pinene in the range of 5.07–6.18%, β -pinene 9.16–11.68%, sabinene 8.5–17.16%, limonene 21.06–22.71% and β -caryophyllene 21.52–27.70%. Zachariah (1995) studied 42 black pepper accessions and reported 3.8–16.6% pinene, 2.2–33% sabinene, 1.6–31.8% myrcene, 3.6–21.2% limonene, 0.2–1.8% linalool and 11.8–41.8% β -caryophyllene.

Orav *et al.* (2004) determined the essential oil composition of black, green and white pepper using GC/mass spectrometry. Most abundant compounds in pepper oils were (E)- β -caryophyllene (1.4–70.4%), limonene (2.9–38.4%), β -pinene (0.7–25.6%), Δ -3-carene (1.7–19.0%), sabinene (0–12.2%), α -pinene (0.3–10.4%), eugenol (0.1–41.0%) terpinene-4-ol (0–13.2%) hedycaryol (0–9.1%), β -eudesmol (0–9.7%) and caryophyllene oxide (0.1–7.2%). Green pepper oil (dried by sublimation method) had a higher content of monoterpenes (84.2%) than air-dried green pepper corns (26.8%). The oil from ground black pepper contained more monoterpenes and less

Table 11.3 Percentage composition of *Ocimum basilicum* volatiles

Compound	% Composition
α -Thujene	0.4
Myrcene	0.4
Limonene + 1,8-cineole	0.4
(E)- β -Ocimene	0.7
γ -Terpinene	0.2
Terpinolene	1.4
β -Elemene	1.2
β -Caryophyllene	0.2
(E)- α -Bergamotene	7.6
α -Caryophyllene	0.4
Germacrene D	0.8
β -Selinene	0.6
Bicyclogermacrene	0.4
γ -Muurolene	0.8
Cadinene	1.4
Cadinol	0.4
p-Cymene	0.5
Estragole	22.2
(Z)-Sabinene hydrate	0.9
Linalool	41.2
Camphor	0.3
Terpin-4-ol	2.3

Source: Sanda *et al.* 1998.

sesquiterpenes and oxygenated terpenoids as compared to green and white pepper oils. Sumathykuty *et al.* (1999) identified elemol as the most abundant component of black pepper leaf oil. Murthy *et al.* (1999) reported that pepper powder with an average particle size of 0.7 mm is essential to release the maximum concentration of monoterpenes and sesquiterpenes.

Jagella and Grosch (1999a), by adopting dilution and concentration experiments as well as enantioselective analysis of optically active monoterpenes, indicated (\pm) linalool, (+)- α -phellandrene, (–)-limonene, myrcene, (–)- α -pinene, 3-methyl butanal and methyl propanal as the most potent odorants of black pepper. Storage studies conducted by Jagella and Grosch (1999b) using ground black pepper revealed that losses of α -pinene, limonene and 3-methyl butanal were mainly responsible for deficits in the pepper-like, citrus-like, terpene-like and malty notes after 30 days at room temperature. The musty/mouldy off flavour of a sample of black pepper was caused by a mixture consisting of 2,3-diethyl-5-methyl pyrazine and 2-isopropyl-3-methoxy pyrazine. The key odorants of white pepper as identified by Jagella and Grosch (1999c) are limonene, linalool, α -pinene, 1,8-cineole, piperonal, butyric acid, 3-methyl butyric acid, methyl propanal and 2- and 3-methyl butanal. Narayanan (2000) described the percentage composition of the volatile constituents in four black pepper varieties Panniyur-1, 2, 3 and 4 (Table 11.4).

Cardamom

The active constituent of cardamom is the aromatic volatile oil. The freshly dried unsplit capsules filled with seeds are the best material for distillation of volatile oil. Oils from freshly separated seeds or from whole capsules are almost identical as the husk practically does not yield any oil (Govindarajan *et al.* 1982). Zachariah (2002) described the chemical composition of cardamom oil from different samples (Table 11.5). Govindarajan *et al.* (1982) described the trace components in cardamom oil (Table 11.6). Gopalakrishnan (1994) conducted studies on the storage quality of CO₂-extracted cardamom oil. The class of components that underwent quantitative reduction was the terpene hydrocarbons in the oil, whereas the other components showed varying responses at low and ambient temperatures of storage

Cassia

Cinnamomum cassia yields bark and leaf oils that are economically important. The bark of *cassia* is coarser and thicker with a more intense aroma than the true cinnamon, *C. verum* (Bercht. and Presl.). The bark is used for flavouring food and beverages and also in pharmaceutical preparations and perfumery. The volatile oils from leaf and bark and the oleoresin from bark are used in soaps, perfumes, spice essences and beverages. The major component of the oil from cassia bark and leaf is cinnamaldehyde.

The *Cinnamomum cassia* Blume bark oil from Nigeria contained mainly cinnamaldehyde, with some eugenol while the leaf oil contained high levels of benzyl benzoate (Lockwood 1979). Cinnamon plants with purple leaf flushes had 29% more bark oil (1.84%) as compared to those with green flushes (1.43%), whereas bark oleoresin (8.41% and 7.90% in purple and green respectively) and leaf oil (1.68% and 1.73% in purple and green respectively) contents were on a par in both the types (Krishnamoorthy *et al.* 1988).

Headspace composition of cinnamon and cassia quills of different origin showed that the cinnamaldehyde and benzaldehyde contents were in the ranges 2.3–86.2% and 0.5–40.5%, respectively (Vernin *et al.* 1994). Jayatilaka *et al.* (1995) examined

Table 11.4 Chemical constituents of four black pepper varieties

No.	Compound	Percentage composition			
		1	2	3	4
1	α -Thujene	0.73	1.26	1.59	0.91
2	α -Pinene	5.28	6.18	5.07	5.32
3	Camphene	0.14	0.18	0.14	0.13
4	Sabinene	8.50	13.54	17.16	1.94
5	β -Pinene	11.08	10.88	9.16	6.40
6	Myrcene	2.23	2.30	2.20	8.40
7	α -Phellandrene	0.68	0.20	–	2.32
8	δ -3-Carene	2.82	0.18	–	1.03
9	α -Terpinene	–	–	0.39	1.13
10	p-Cymene	–	0.18	0.07	9.70
11	(Z)- β -Ocimene + β -phellandrene	–	0.15	0.23	0.37
12	Limonene	21.06	21.26	22.71	16.74
13	(E)- β -Ocimene	0.18	2.84	0.30	0.17
14	γ -Terpinene	0.01	0.49	–	0.03
15	<i>Trans</i> -sabinene hydrate	0.14	–	0.30	0.19
16	Terpinolene	0.10	0.20	0.22	0.08
17	<i>Trans</i> -linalool oxide	0.03	0.18	–	0.08
18	Linalool	0.22	0.22	0.46	0.28
19	<i>Cis</i> -p-menth-2-en-1-ol- + <i>cis</i> -p-menth-2,8-diene-1-ol	0.04	0.04	0.05	0.02
20	<i>Trans</i> -p-menth-2-en-1-ol	0.01	0.01	0.01	0.01
21	Citronellal	0.02	0.03	0.03	0.01
22	p-Menth-8-en-1-ol	0.03	t	–	t
23	Borneol	t	t	t	t
24	Terpinen-4-ol	0.19	0.32	0.52	0.18
25	α -Terpineol	0.10	0.17	0.12	0.07
26	Dihydrocarveol	0.01	–	0.02	0.02
27	p-Menth-8-en-2-ol	–	0.01	0.02	0.02
28	<i>Trans</i> -carveol	0.01	0.01	–	0.02
29	<i>Cis</i> -carveol + carvone	0.01	0.03	0.03	0.03
30	Piperitone	0.04	t	0.03	t
31	Carvone oxide	0.01	0.01	–	0.01
32	Myrtenol	0.20	0.04	0.11	0.04
33	α -Terpinyl acetate	0.86	1.22	1.33	1.05
34	Neryl acetate	0.20	0.07	0.05	0.13
35	Geranyl acetate	0.12	0.01	0.07	0.11
36	α -Cubebene/ δ -elemene	3.25	0.26	0.16	2.56
37	α -Copaene	0.82	0.49	0.44	0.71
38	β -Elemene	0.09	0.09	0.06	0.05
39	β -Caryophyllene	21.59	27.70	23.2	21.19
40	<i>Trans</i> - α -berga motene	0.31	–	–	0.28
41	α -Humulene	0.21	0.20	–	0.29
42	(E)- β -Farnesene	0.08	0.22	0.11	0.13
43	α -Amorphene	1.51	1.53	0.03	1.28
44	α -Guaiene	0.11	0.07	1.54	0.10
45	Clovene	0.14	0.07	0.07	0.13
46	Germacrene-D	0.04	0.03	0.04	0.26
47	ar-curcumene	0.26	0.12	0.04	0.29
48	β -Selinene	0.64	0.87	1.37	0.63
49	α -Selinene	0.07	0.12	0.48	0.14
50	δ -Murolene	0.73	0.93	0.16	0.58
51	(E,E)-2-Farnesene	0.72	–	0.47	0.72

Table 11.4 Continued

No.	Compound	Percentage composition			
		1	2	3	4
52	β -Bisabolene +2-bisabolene	4.25	2.15	3.10	0.49
53	δ -Guaiene	0.82	0.17	0.09	1.85
54	Cuparene	1.38	0.09	0.14	0.04
55	δ -Cadinene	0.12	–	0.07	0.13
56	(2)-Nerolidol	0.20	0.05	0.11	0.05
57	Elemol	0.11	0.06	0.07	0.08
58	(E)-Nerolidol	0.12	0.04	0.07	0.03
59	Caryophyllene alcohol	0.07	0.02	0.04	0.02
60	Caryophyllene oxide	0.90	0.35	0.38	0.25
61	Cedrol	0.07	–	0.05	0.05
62	α -Cadinol	1.51	0.29	0.12	1.27
63	α -Cadinol	0.26	0.12	0.15	0.25
64	β -Bisabolol	0.20	0.09	0.17	0.14

Source: Narayanan 2000.

t = trace (<0.01%)

1 = Panniyur-1 2 = Panniyur-2 3 = Panniyur-3 4 = Panniyur-4.

Table 11.5 Percentage composition of cardamom volatile from different sources

Component	Var. Malabar (Ceylon)	Var. Malabar (Gautemala)	Var. Mysore	Sri Lanka (Wild)
α -Pinene	1.10	0.71	1.40	13.00
Camphene	0.02	0.03	0.04	0.13
Sabinene	2.50	3.40	3.10	4.90
β -Pinene	0.20	0.34	0.26	4.90
Myrcene + terpinene	1.80	1.50	1.10	2.50
α -Phellandrene	<0.01	<0.01	<0.01	0.42
D-Limonene	0.02	0.12	0.14	2.10
1,8-Cineole	31.0	23.4	44.0	3.30
γ -Terpinene	0.12	0.34	0.10	22.2
Linalool	2.10	4.50	3.00	3.70
Citronellal	<0.01	0.04	0.06	0.13
4-Terpineol	0.14	0.28	0.87	15.3
α -Terpineol	1.40	1.90	1.50	0.86
Citronellol	<0.01	0.04	<0.01	0.01
Nerol	0.02	0.04	0.06	0.78
Linalyl acetate	3.30	6.30	3.10	0.31
Geraniol	0.27	0.38	0.25	0.34
α -Terpinyl acetate	52.5	50.7	37.0	0.14
Geranyl acetate	0.08	0.13	0.15	1.50
<i>Trans</i> -nerolidol	0.09	0.83	0.07	0.44
<i>Cis</i> -nerolidol	0.23	1.60	0.28	0.37

Source: Zachariah 2002.

the composition of bark oil from 25 samples of *Cinnamomum cassia* and the major components identified were (E)-cinnamaldehyde (92.0–98.0%), (Z)-cinnamaldehyde (0.8–2.7%), β -caryophyllene (0.4–3.6%), coumarin (0.1–1.6%) and α -ylangene (0.1–2.7%). Analysis of the chinese cassia oil by HPLC method and supercritical CO₂ extraction indicated cinnamaldehyde content as 68.2–71.9% and 73.9–74.4%,

Table 11.6 Trace components in cardamom volatile oil

Hydrocarbons	Alcohols and phenols
α -Thujene	3-Methyl butanol
Camphene	<i>p</i> -Menth-3-en-1-ol
α -Terpinene	Perillyl alcohol
<i>cis</i> -Ocimene	Cuminy alcohol
<i>trans</i> -Ocimene	<i>p</i> -Cresol

Source: Govindarajan *et al.* 1982.

respectively (Ehlers *et al.* 1995, Lawrence 2001). Evaluation for chemical constituents in open pollinated seedling progenies of *C. cassia* accessions from Calicut (India) showed that these contained 1.2–4.95% bark oil, 6.0–10.5% bark oleoresin and 0.4–1.65% leaf oil. The major component of both the oils, namely, cinnamaldehyde, varied from 40.7–86.0% and 61.9–91.5% respectively in leaf and bark oils (Krishnamoorthy *et al.* 1999). The leaf oil of cassia from China contained 74.1% cinnamaldehyde, 10.5% 2-methoxy cinnamaldehyde and 6.6% cinnamyl acetate as major components whereas the Australian cassia recorded 77.2% cinnamaldehyde, 15.3% coumarin and 3.6% cinnamyl acetate as chief constituents (Dao 1999). Composition of leaf and bark oil of *Cinnamomum cassia* from Yunnan Province is indicated in Table 11.7 (Li *et al.* 1998).

Cinnamon

Cinnamomum verum (Syn. *C. zeylanicum*) yields mainly leaf and bark oils, that are used in perfumery and flavouring. The major component of the leaf oil is eugenol while that of bark oil is cinnamaldehyde. Senanayake *et al.* (1978) identified 32 components in cinnamon oil, of which eugenol (70.1%) and cinnamaldehyde (75.0%) were the major compounds in leaf and bark respectively. The oil from its root bark contained camphor (56.2%) and 1,8-cineole (11.7%) as chief components. The cinnamon varieties Navashree and Nithyasree, recorded 2.7–2.8% bark oil, 10% bark oleoresin and 3% leaf oil contents (Krishnamoorthy *et al.* 1996). Two types of *Cinnamomum zeylanicum* leaf oils exist, the main constituent of one being eugenol and that of the other benzyl benzoate. Nath *et al.* (1996) reported a variety of *C. verum* growing in Brahmaputra valley (India) with benzyl benzoate as a major constituent in both leaf and bark oils. The essential oil of the leaves of *C. zeylanicum* from Cameroon contained eugenol (85.2%), (E)-cinnamaldehyde (4.9%), linalool (2.8%) and β -caryophyllene (1.8%) (Jirovetz *et al.* 1998).

A chemotype of *Cinnamomum zeylanicum* with 85.7% linalool in leaf oil was reported from Calicut (South India) by Jirovetz *et al.* (2001) (Table 11.8). Cinnamon leaf oils of Indian origin contained 81.43–84.5% eugenol (Mallavarappu *et al.* 1995) (Table 11.9). Syamasundar *et al.* (2000) reported variation in the composition of unripe and ripe fruits of cinnamon. The oil from unripe fruits was dominated by δ -cadinene (19.15%), α -pinene (11.47%), β -pinene (10.51%), E-cinnamyl acetate (7.11%) and γ -cadinene (8.05%) whereas the ripe fruits contained γ -cadinene (23.48%), α -pinene (11.52%), E-cinnamyl acetate (8.62%) and α -muurolene (8.22%) as chief components. The fruit oil from South India was dominated by α -pinene (11.2%), β -pinene (9.2%), β -caryophyllene (11.0%), α -muurolene (6.1%), δ -cadinene (20.2%) and α -muurolol (9.8%) (Mallavarapu and Ramesh 2000) (Table 11.10). Volatile oil from cinnamon flowers was dominated by (E)-cinnamyl acetate (41.98%), trans- α -

Table 11.7 Comparative percentages, composition of the leaf and bark oils of *Cinnamomum cassia*

Compound	Leaf oil (%)	Bark oil (%)
α -Pinene	0.05–0.36	0.10–0.25
Camphene	0.04–0.05	0.05–0.10
β -Pinene	0.04–0.15	0.14–0.22
Myrcene	0.02–0.03	t–0.10
α -Phellandrene	0.01–0.03	t–0.13
Limonene	0.13–0.24	0.14–0.29
1,8-Cineole	0.05–0.08	0.06–1.07
δ -3-Carene	0.03–0.05	t–0.07
<i>p</i> -Cymene	0.11–0.19	0.04–0.18
Camphor	0.07–0.15	0–0.08
Benzaldehyde	1.42–1.48	0.50–1.10
Linalool	0.11–0.23	0.08–0.16
Terpinolene	t	0–0.04
β -Caryophyllene	0.16–0.20	t–0.27
α -Humulene	t–0.03	0–0.15
β -Elemene	–	t–0.06
Isoborneol	0–0.20	0–0.27
Borneol	0.15–0.41	0.06–1.27
α -Terpineol	t–0.10	0.07–2.05
Geraniol	t	0.08–0.31
Carvone	0.57–0.64	0–0.34
2-Methoxybenzaldehyde	0.08	0–0.12
Safrole	–	t–0.20
γ -Elemene	0–t	0–0.41
δ -Cadinene	t	t–0.13
β -Cadinene	–	t–0.10
Hydrocinnamaldehyde	0.88–0.89	0–0.24
Phenylacetaldehyde	0.07–0.16	t–0.27
Methyl eugenol	0.14–0.15	t–0.05
(E)-Cinnamaldehyde	64.10–68.30	80.40–88.50
α -Copaene	0.41–0.49	0.23–0.68
Vanillin	t	t–0.10
Salicylaldehyde	0.05–0.42	0.04–0.85
2-Phenethyl alcohol	0.11–0.27	t–0.16
Benzyl alcohol	t–0.05	–
Acetophenone	t–0.1	0–0.6
Eugenol	0.04–0.06	0.03–1.08
(Z)-Isoeugenol	0.14–0.28	0.12–0.66
(E)-Cinnamyl acetate	4.50–12.50	0.60–5.10
γ -Muurolene	t	t–0.50
Anisaldehyde	0.58–1.02	t
2-Phenethyl acetate	t–1.55	–
β -Bisabolene	t–0.06	t–0.18
β -Bisabolol	t	t–0.35
α -Muurolol	0–0.08	0–0.24
Coumarin	0.03–0.08	t–0.45
(E)-Cinnamic acid	0.80–2.48	0.12–3.10
(E)-2-Methoxycinnamaldehyde	8.40–10.50	t–2.50
Hydrocinnamic acid	0.18–0.51	0.024
4-Hydroxy-2-phenethyl alcohol	0–0.12	0–0.10
Caryophyllene oxide	0.15–0.17	0–0.10
Patchoulene	0.06–0.07	0–0.04
Octanoic acid	t	0–t

Table 11.7 Continued

Compound	Leaf oil (%)	Bark oil (%)
3-Phenylpropyl acetate	0.21–0.43	0.05–0.22
Nonanoic acid	t–0.10	0–t
Guaicol	t	0–0.08
(E)-Cinnamyl alcohol	0.15	0.05–0.13
(E)-Ethyl cinnamate	0.11–0.27	t–0.14
Benzyl benzoate	0.07–0.15	t–0.38
Methyl alaninate	t–0.05	–
Guaicyl cinnamate	t	t
Decanoic acid	t	0–t
Undecanoic acid	0–0.05	0–0.11
Dodecanoic acid	t–0.04	0–t
Benzoic acid	0.07–0.11	0.07–0.10
Salicylic acid	t–0.10	0.10–0.20

Source: Li *et al.* 1998.

Note: T = trace.

Table 11.8 Composition of oil from *Cinnamomum zeylanicum* leaves from Calicut, India

(E)-2-Hexenol (0.1%)	Borneol (0.1%)
(Z)-3-Hexenol (0.1%)	Terpinen-4-ol (0.3%)
1-Hexen-3-ol (0.1%)	α -Terpineol (1.1%)
Hexanol (0.1%)	Dihydrocarveol (t)
α -Pinene (t)	Linalyl acetate (0.1%)
(Z)-3-Hexenyl acetate (0.1%)	(E)-Cinnamaldehyde (1.7%)
(E)-2-Hexenyl acetate (0.1%)	Safrole (t)
p-Cymene (t)	(E)-Cinnamyl alcohol (0.1%)
β -Phellandrene (t)	Eugenol (3.1%)
(E)- β -Ocimene (t)	(E)-Cinnamyl acetate (0.9%)
1,8-Cineole (0.1%)	β -Caryophyllene (2.4%)
Limonene (0.2%)	α -Humulene (0.2%)
<i>Cis</i> -Linalool oxide* (0.1%)	Eugenyl acetate (0.1%)
Terpinolene (0.1%)	Caryophyllene oxide (0.1%)
<i>Trans</i> -Linalool oxide* (0.1%)	Spathulenol (0.2%)
Linalool (85.7%)	
Nonanol (0.3%)	

Source: Jirovetz *et al.* 2001.

Note: * furanoid form; t = trace (<0.01%).

bergamotene (7.97%), caryophyllene oxide (7.29%) and α -cadinol (6.35%) (Jayaprakasha *et al.* 2000).

Clove

Clove essential oils are extracted from *Eugenia caryophyllata* (*Syzygium aromaticum*, *Eugenia aromatica*, *E. caryophyllus*) from the Myrtaceae family. Clove oil is extracted from the leaves, stem and buds. However, only the clove bud oil is used in aromatherapy, since it contains less eugenol. Phenolic reactivity was seen almost throughout the bud, with a greater concentration in the outer glandular region of the hypanthium than in the inner aerenchymatous spongy tissue (Mangalakumari and Mathew 1985). Dried leaves of clove grown in Little Andaman (India), on hydrodistillation, gave

Table 11.9 Volatiles from *Cinnamomum verum* leaves

Compound	% Composition
	Leaf
α -Thujene	0.04–0.06
α -Pinene	0.38–0.49
Camphene	0.17–0.18
Sabinene	t
α -Pinene	0.16–0.18
Myrcene	0.09–0.13
n-Octanal	t
α -Phellandrene	0.50–1.03
Δ -3-Carene	0.05
α -Terpinene	0.03
p-Cymene	0.16–0.28
1,8-Cineole	0.23–0.38
β -Phellandrene	t
(z)- β -Ocimene	t
(E)- β -Ocimene	0.05
γ -Terpinene	0.05
<i>cis</i> -Linalool oxide (furanoid)	t
<i>trans</i> -Linalool oxide (furanoid)	t
	0.05–0.11
Terpinolene	1.57–3.70
Linalool	t
2-Phenylethanol	0.10
Camphor	0.39
Citronellal	0.12
Borneol	0.04–0.05
Terpinen-4-ol	0.10–0.14
α -Terpineol	0.19
Methylchavicol	t
(Z)-Cinnamaldehyde	0.26
Nerol	t
Cuminaldehyde	0.03
Piperitone	0.63–1.51
(E)-Cinnamaldehyde	0.52
Linalyl acetate	0.19
Safrole	t
(E)-Cinnamyl alcohol	t
2-Phenylethyl propionate	81.43–84.50
Eugenol	t
(E)-Methyl cinnamate	t
(Z)-Cinnamyl acetate	0.25–0.28
β -Elemene	t
(Z,E)- α -Farnesene	0.73
(E)-Cinnamyl acetate	2.49
β -Caryophyllene	0.47–2.25
(Z)-Methyl isoeugenol	t
α -Humulene	0.12–0.46
(E)-Methylisoeugenol	t
β -Selinene	t
Eugenyl acetate	0.14–2.85
γ -Cadinene	

Source: Mallavarapu *et al.* (1995).

Note: t = trace (<0.01%).

Table 11.10 Composition of *Cinnamomum zeylanicum* fruit oil

(E)-2-Hexenol (t)	(E)-Cinnamyl acetate (0.4%)
Tricyclene	β -Caryophyllene (11.0%)
α -Pinene (11.2%)	(E)- β -Farnesene (0.8%)
Camphene (0.6%)	α -Humulene (2.2%)
β -Pinene (9.2%)	γ -Muuroolene (0.2%)
Myrcene (1.6%)	Germacrene D (0.2%)
α -Phellandrene (0.7%)	α -Muuroolene (6.1%)
α -Terpinene (0.2%)	δ -Cadinene (7.1%)
p-Cymene (0.1)	δ -Cadinene (13.1%)
Limonene (2.8%)	Cis-Calaminnene (2.2)
1,8-Cineole (0.1%)	α -Cadinene (1.2%)
(Z)- β -Ocimene (0.1%)	Elemol (1.9%)
(E)- β -Ocimene (0.2%)	(E)-Nerolidol (0.1%)
γ -Terpinene (0.1%)	Isocaryophyllene oxide (0.2%)
Tepinolen (0.5%)	Spathulenol (0.8%)
Linalool (0.2%)	Caryophyllene oxide (0.4%)
α -Fenehyl alcohol (0.5%)	Globulol (0.4%)
Isoborneol (t)	Humulene epoxide I (0.5%)
Borneol (0.5%)	Humulene epoxide II (0.6%)
Terpinen-4-ol (0.1%)	1-Epi-cubenol (0.1%)
α -Terpineol (0.5%)	T-Cadinol (0.2%)
Nerol (t)	Cubenol (0.9%)
Geraniol (t)	α -Muurolol (9.8%)
Isobornyl acetate (0.1%)	Selin-11-en-4a-ol (0.1%)
(Z)-Cinnamyl acetate (0.1%)	α -Cadinol (3.1%)
α -Copaene (2.1%)	4-Hydroxy-3,4-dihydrocalacorene* (0.2%)
β -Elemene (0.4%)	4-Hydroxy-3,4-dihydrocalacorene* (0.1%)

Source: Mallavarapu and Ramesh, 2000.

Notes: * correct isomer not identified; t = trace (<0.1%).

4.8% oil. The major compound was eugenol (94.4%), followed by β -caryophyllene (2.9%) (Raina *et al.* 2001) (Table 11.11).

The chemical composition of bud and leaf oils of *S. aromaticum* from Cuba indicated 36 and 31 volatile compounds, respectively. The major components of the bud oil were eugenol (69.8%), β -caryophyllene (13.0%) and eugenyl acetate (16.1%), whereas the leaf oil contained eugenol (78.1%) and β -caryophyllene (20.5%) as the main constituents (Pino *et al.* 2001). During leaf growth (between days 2 (initial leaf stage) to 41 (yellow leaves) days), the content of caryophyllene in the essential oil of leaves decreased from 6.3% to 0.2% and the content of eugenol acetate decreased from 51.2% to 1.5% but the eugenol content increased from 38.3% to 95.2% (Gopalakrishnan and Narayanan 1988).

In the clove bud and stem essential oils from Madagascar four components predominated: eugenol (73.5–79.7% in bud and 76.4–84.8% in stem oils); β -caryophyllene (7.3–12.4% in both oils); α -humulene (1.0–1.4% in both oils); and eugenyl acetate (4.5–10.7% and 1.5–8.0%, respectively) (Gaydou and Randriamiharisoa 1987). The neutral fraction of the bud oil from Madagascar contained β -caryophyllene (75.64%), α -humulene (14.12%) and δ -cadinene (2.34%) as the major components (Muchala and Crouzet 1985). Gopalakrishnan and Narayanan (1988) reported that the eugenol content in leaves increased from 38.3% to 95.2%, with maturity, while the contents of eugenyl acetate (51.2% to 1.5%) and caryophyllene (6.3% to 0.2%) decreased. The clove bud and stem oils from Madagascar were dominated by eugenol, eugenyl acetate and β -caryophyllene (Gaydou and Randriamiharisoa 1987).

Table 11.11 Percentage composition of clove oil

Components	Percentage
(E)- β -Ocimene	0.03
Linalool	0.08
Terpinen-4-ol	0.03
Nerol	0.79
Eugenol	94.4
α -Copaene	0.04
β -Caryophyllene	2.91
α -Humulene	0.36
(E,E)- α -Farnesene	0.06
γ -Cadinene	0.18
(E)-Nerolidol	0.03
β -Caryophyllene oxide	0.67
Humulene oxide II	0.07
l-Cadinol	0.07
Cadalene	0.18
Hexadecyl acetate	0.09

Source: Raina *et al.* (2001).

Gopalakrishnan *et al.* (1984) characterized six sesquiterpenes namely, α -cubebene (1.3%), α -copaene (0.4%), α -humulene (9.1%), β -caryophyllene (64.5%), γ -cadinene (2.6%) and δ -cadinene (2.6%) in the hydrocarbon fraction of the freshly distilled Indian clove bud oil. Clove oil from the Malagasy republic was dominated by eugenol (72–73%), eugenyl acetate (6.3–7.8%) and caryophyllene (15.7%) (Lawrence and Reynolds 1985). The essential oil content ranged from 12.9–18.5% in clove buds and 3.0–7.7% in pedicel. Eugenol content varied from 44–55% in bud oil and 60.0–72.4% in the oil from pedicel (Zachariah *et al.* 2005).

Coriander

Coriander oil is clear, colourless to light yellow liquid. Norwegian seeds contain higher levels of volatile oil (1.4–1.7%) (Purseglove *et al.* 1981b). Indian coriander seeds are poor in oil content (0.1–0.4%) (Agrawal and Sharma 1990). The major component of the essential oil was linalool (67–70%). Kumar *et al.* (1977) observed that small-fruited coriander was characterized by high oil content and preferred for distillation. Large fruited coriander seeds are lower in oil content and are more suited for use as spice.

Leaf oil of coriander is dominated by decanal (10%) and dodecanals (35%). Indian coriander oil is lower in linalool content and higher in linalyl acetate (Rao *et al.* 1925). Coriander seed oil contained 21% linalyl acetate and 42% linalool (Gupta *et al.* 1977). Steam distilled oil contained less linalool (71.9%) compared to CO₂ extract (83.2%) (Hirvi *et al.* 1986). Boelens *et al.* (1989) reported that linalool content (70.4%) was higher by hydrodistillation as against by hydrodiffusion (66.2%) and organoleptic preference was slightly more for the oil obtained by hydrodiffusion over hydrodistillation.

Nitz *et al.* (1992) compared the composition of the distilled oil of coriander with that of the SFE extract and found that the major compounds were linalool (63%), limonene (4%), γ -terpinene (9%), camphor (4%), α -pinene (8%) and geranyl acetate (2%). Diederischen (1996) analyzed 237 accessions of fruit oil and the main constituents

in these varied as follows: α -pinene (6.5–28.9%), γ -terpinene (0.7–35.4%), camphor (0.4–6.3%), linalool (19.8–82.0%), geranyl acetate (1.3–12.4%) and geraniol (0.3–3.3%).

Bandoni *et al.* (1998) compared the composition of coriander seed oil produced by water and steam distillation and found that the oils were quite similar. The chemical composition of the seed essential oil grown in Brazil contained linalool (77.48%), γ -terpinene (4.64%), α -pinene (3.97%), limonene (1.28%), geraniol (0.64%) and 2-decenal (0.16%) as the main components (Figueiredo *et al.* 2004).

Cumin

Cumin seeds yield 2.3–4.8% volatile oil. The oil is yellow amber liquid that tends to darken on ageing. The characteristic odour of cumin is mainly due to the aldehydes present in the seeds namely, cuminaldehyde, p-menth-3-en-7-al and p-menth-1,3-dien-7-al. (Agrawal 2001). Indian cumin oil is reported to be lower in cuminaldehyde content. Turkish cumin seed oil was reported to have cuminaldehyde (19.2%), p-mentha-1,3-dien-7-al (4.2–12.2%), p-mentha-1,4-dien-7-al (24–48%), γ -terpinene (7.0–14.1%), p-cymene (9.1–12.0%) and β -pinene (2.9–8.9%) as major constituents (Baser *et al.* 1992). Shaath and Azzo (1993) reported 25.01% cuminaldehyde in the cumin seed oil of Egyptian origin (Table 11.12). Pande and Goswami (2000) identified 12 constituents contributing to 86.4% of the oil of which the chief components were cuminaldehyde (32.6%), p-cymene (14.7%), p-mentha-1,4-dien-7-al (13.5%) and β -pinene (12.7%).

Dill

Essential oil is extracted from the seeds and leaves of dill. Fresh herb yields 0.19% light yellow oil and seeds yield 1% oil (light yellow). The major component of seed oil is d-carvone while that of leaf oil is α -phellandrene (Guenther 1961a, Pino *et al.* 1995, Kruger and Hammer 1996, Faber *et al.* 1997, Ranade 1998, Vera and Chanem-Ming 1998 and Minija and Thoppil 2004).

Ravid *et al.* (1987) isolated optically active S (+)-carvone, the major component of the fruits of dill oil. The importance of S-(+) carvone is that it is used as the starting material for the synthesis of (R, Z)-3-methyl-6-isopropenyl-3,9-decadien-1-yl acetate, a pheromone component of the female California red scale, while R(-)-carvone is used as a starting material in the preparation of picrotoxinin (Ravid *et al.* 1987).

Table 11.12 Chemical composition of cumin seed oil of Egyptian origin

Compound	Percentage content
α -Thujene (0.28%)	Terpinolene (0.11%)
α -Pinene (0.78%)	Terpinen-4-ol (0.16%)
Camphene (trace)	p-menth-3-en-7-al (3.83%)
Sabinene (0.40%)	α -terpineol (0.05%)
β -Pinene (14.64%)	Cuminaldehyde (25.01%)
Myrcene (0.92%)	p-mentha-1,4,dien-7-al (17.36%)
α -Phellandrene (0.63%)	p-mentha-1,3,dien-7-al (5.84%)
p-Cymene (4.91%)	β -caryophyllene (0.20%)
β -Phellandrene (0.30%)	<i>Trans</i> - α -bergamotene (0.31%)
limonene (0.37%)	
γ -Terpinene (19.12%)	

Source: Shaath and Azzo (1993).

Huopalahti *et al.* (1988) compared the composition of dill herb oil obtained by hydrodistillation, solvent extraction and CO₂ extraction by GC-MS and HS-GC (head-space GC). Each method gave different composition for the volatiles of dill herb (Table 11.13). However, the maximum concentration of the most important aroma compound in the dill herb, namely, 3,6-dimethyl-2,3,3a,4,5,7a-hexahydro benzofuran was obtained with head-space GC (38.5% \pm 1.2%) analysis. The oil obtained by the hydrodistillation method contained 3,6-dimethyl-2,3,3a,4,5,7a-hexahydrobenzofuran (36.7% \pm 1.6%) and α -phellandrene (32.1% \pm 1.6%) as major components. CO₂ extracted oil contained 3,6-dimethyl-2,3,3a,4,5,7a-hexahydrobenzofuran (33.2% \pm 3.7%) and neophytadiene (19.9% \pm 4.3%). The solvent extracted oil was dominated by 3,6-dimethyl-2,3,3a,4,5,7a-hexahydro benzofuran (27.1% \pm 2.2%), α -phellandrene (22.3% \pm 3.7%) and neophytadiene (14.0% \pm 1.8%).

Pure oil of dill weed should contain a minimum of 5% 3-9-epoxy-p-menthene. In pure dill oil the percentage ratios of α -phellandrene to limonene to β -phellandrene are 20:25:3. The chemical composition of dill oil of Hungarian origin is as follows: α -thujene (0.3%), α -pinene (0.8%), myrcene (0.7%), α -phellandrene (29%), limonene (25%), β -phellandrene (4.2%), p-cymene (1.3%), 1-methyl-4-isopropyl-benzene, α -p-dimethylstyrene (trace), dihydrocarvone (0.3%), isodihydrocarvone (0.2%), and D-carvone (35.2%). Lab distilled oil of *Anethum graveolens* seed from Pakistan indicated the presence of limonene (9.34%), dillapiole (28.28), carvone (52.25%) and dihydrocarvone. (Lawrence 1981). Zawirska-Wojtasiak *et al.* (1998) studied the aroma profile of dill varieties grown in Poland. They found that carvone and limonene amount to 90–96% of total volatiles content. Other compounds of the oil are α -pinene, α -phellandrene, p-cymene, terpinene-4-ol, dihydrocarvone, eugenol and vanillin. They could establish variability in the organoleptic properties between varieties.

Fennel

Fennel seeds yield about 2–2.5% oil on dry weight basis. Fennel seeds have fragrant odour and pleasant aromatic taste. There are two types of fennel – common fennel and sweet fennel. Common fennel (*Foeniculum vulgare* Mill) contains 2.5–6.5% volatile oil. The oil is a colourless to pale yellow liquid with an aromatic, spicy

Table 11.13 Composition of dill volatiles extracted by different methods

No.	Compound	Amounts (%)			
		Solvent extraction	Hydro-distillation	CO ₂ -extraction	Head-space
1.	α -Pinene	0.5	1.1	0.2	1.4
2.	α -Phellandrene	22.3	32.1	6.8	16.1
3.	Limonene	1.5	2.5	0.7	3.2
4.	β -Phellandrene	3.7	5.6	1.7	5.5
5.	p-Cymene	2.1	5.7	1.8	13.5
6.	3,6-Dimethyl-2,3,3a,4,5,7a-hexahydrobenzofuran	27.1	36.7	33.2	38.5
7.	Carvone	0.7	0.8	1.0	t
8.	Neophytadiene	14.0	tr	19.9	
9.	Myristicin	3.3	2.9	5.8	4.7
10.	Apiol	3.1	0.6	4.2	

Source: Huopalahti *et al.* (1988).

odour. The major component in the seed oil is anethole. The herb oil of fennel contains α -phellandrene, pinenes, anethole and methyl chavicol. Bitter fennel oil is obtained from *F. vulgare* var. *vulgare* which is cultivated in Europe. Sweet fennel (*F. vulgare* Mill var. *dulce*) is mainly cultivated in France and Italy. It is also known as Roman or French oil. The essential oil is yellowish green liquid with characteristic Anise odour.

Naves and Tucakov (1959) reported that Yugoslavian fennel oil contained *trans*-anethole (50–80%), *cis*-anethole (>0.3%), methyl chavicol (3–20%) and fenchone (0.7–2.2%). Indian fennel oil was found to contain 1,8-cineole (1.95%), linalool (7.98%), safrole (3.67%), anisaldehyde (8.72%), anethole (64.88%) and methyl chavicol (1.94%) (Srinivas 1986, Raina *et al.* 2004). The main constituents are anethole (50–60%) and fenchone (10–25%) (Agrawal 2001). Yamini *et al.* (2002) compared the compositions of hydrodistilled and supercritical CO₂ extracted oils from the fennel seeds from Iran with those of France and Spain. Both contained anethole as the major component, but at higher temperatures and pressures higher solubility of anethole was noticed (Table 11.14). The major compounds in the oils from Iran and Spain contained anethole and limonene, but the oil from Iran was richer in E-anethole whereas the Spanish oil contained relatively higher amount of limonene. The oil from France was markedly different from both these oils. The French oil was dominated by limonene with traces of E-anethole.

Fenugreek

Fenugreek has been used in Indian folk medicine as an antipyretic, diuretic and suppurative and for treatment of dropsy, heart disease, chronic cough and spleen and liver enlargement (Bhatti *et al.* 1996). Studies on the effect of roasting on the quality of fenugreek seeds indicated that light roasted seeds (150 °C) were superior to those roasted at 175 °C and 200 °C with respect to their flavour (Sankaracharya *et al.* 1973).

Girardon *et al.* (1989) identified 39 components including n-alkanes, sesquiterpenes and some oxygenated compounds in the volatiles of fenugreek. But 3-hydroxy-4,5-dimethyl-2(5H)-furanone, which was earlier proposed as a flavouring component of fenugreek seeds was not identified in the volatiles by Girardon *et al.* (1989). However, the contribution of n-alkanes to the aroma of fenugreek seeds was considered minimal. According to Girardon *et al.* (1989) elemenes, muurolens and γ - and δ -lactones that are present in small quantities could be of great importance in the aroma of seeds because of their olfactory properties. Compared to volatile oil, solvent extracts of fenugreek gave typical flavour of fenugreek and the characteristic compound was identified as 3-hydroxy-4,5-dimethyl-2(5H)-furanone (Girardon *et al.* 1989).

Fresh aerial parts of fenugreek plant yielded 0.3% light yellow oil. The main constituents of the oil were δ -cadinene (27.6%), α -cadinol (12.1%), γ -eudesmol (11.2%) and α -bisabolol (10.5%). Other constituents were α -muurolene (3.9%) liguloxide (7.9%), cubenol (5.7%), α -muurolol (4.2%) and epi- α -bisabolol (5.7%) (Ahmadiani *et al.* 2004).

Ginger

Ginger is valued primarily for its aroma and in some products for its mild pungency. These characters together contribute to the typical ginger flavour. Ginger oil is prepared by steam distillation and the aroma quality and composition will depend on the raw material and the area of cultivation. Agroclimatic conditions play a great role in the

Table 11.14 Volatiles from fennel

Compound	% Composition
α -Pinene	0.76–2.00
Camphene	0.09
Sabinene	0.15–0.89
β -Pinene	0.36
Myrcene	0.58–2.24
α -Phellandrene	0.8–7.67
ρ -Cymene	0.24
Limonene	2.82–52.4
(z)- β -Ocimene	0.79–1.39
γ -Terpinene	0.09–12.1
Fenchone	0.3–11.00
Terpinolene	0.13
Camphor	0.24–0.62
Linalyl propanoate	0.32
Estragole	0.78–4.45
ρ -Anisaldehyde	0.57–2.8
(Z)-Anethole	0.27
(E)-Anethole	0.4–90.14
Copaene	0.1
Germacrene D	0.25–5.26
δ -3-Carene	0.1–0.3
α -Terpinene	0.1
l-Limonene	2.1–3.7
l-Fenchone	7.0–11.6
Methyl chavicol	3.1–10.8
t-Carveol	0.1
carvone	tr
α -Fenchyl acetate	0.1
Safrole	tr
Trans-anethole	73.2–80.4
α -Copaene	tr
n-Tetradecane	0.2–0.9
Anisketone	0.2–0.9
n-Hexadecane	0.2
Dillapiole	0.1
Apiole	tr

Source: Yamini *et al.* (2002).

concentration of these constituents. Dry ginger oil is characterized by the high proportion of sesquiterpene hydrocarbons, predominantly zingiberene, a small percentage of monoterpene hydrocarbons and oxygenated compounds (Govindarajan 1982).

Nishimura (2001) separated odorants from fresh rhizomes of Japanese ginger using the multidimensional GC system and found that monoterpenoids such as linalool, 4-terpineol, isoborneol, borneol, geranial and neral contribute towards the characteristic odour. Bartley and Jacons (2000) described the ginger volatiles from fresh and dry rhizomes. The oil is extracted using supercritical carbon dioxide (Table 11.15). Vernin and Parkanyi (2005) compared chemical composition of commercial oils from India and China. Zingiberene and ar-curcumene levels are on a par in both types.

Mint

Japanese mint (*Mentha arvensis*) popularly known as menthol mint is a source of natural menthol which is widely used in pharmaceutical and flavour industries. Xue-

Table 11.15 Volatile compounds in supercritical fluid extracts of fresh and dried ginger

Compound	Amount fresh (%)	Amount dry (%)
Octane	0.39	0.07
Hexanal	1.58	0.87
α -Pinene	0.35	1.24
Camphene	1.08	2.89
β -Pinene	0.00	0.11
6-Methyl-5-hepten-2-one	0.00	0.04
β -Myrcene	0.30	0.94
Octanal	0.42	0.24
Octan-2-ol	0.13	0.31
Limonene	0.27	0.31
β -Phellandrene	1.30	4.68
Heptyl acetate	0.00	0.11
Terpinolene	0.00	0.12
Linalool	0.41	0.39
Citronellal	0.02	0.14
Isoborneol	0.00	0.11
Borneol	0.73	0.39
Decane	0.00	0.00
Decanal	0.96	0.91
Citronellol	0.76	0.47
Neral	1.46	2.30
Geraniol	3.11	1.14
Geranial	18.47	3.90
Bornyl acetate	0.00	0.04
2-Undecanone	0.11	0.24
Citronellyl acetate	0.47	0.77
α -Camphane	0.00	0.17
Geranyl acetate	3.00	5.87
δ -Elemene	0.43	0.60
β -Elemene	0.00	0.14
γ -Elemene	0.16	0.30
(Z)- β -Farnesene	0.15	0.31
(E)- β -Farnesene	0.14	0.17
α -Guaicene	0.02	0.21
ar-Curcumene	1.54	2.29
Germacrene D	0.74	1.26
Zingiberene	13.44	24.58
(E,E)- γ -Farnesene	7.13	14.19
β -Bisabolene	2.49	3.32
γ -Cadinene	0.22	0.19
β -Sesquiphellandrene	5.85	7.64
Elemol	0.80	0.44
Nerolidol	0.38	0.38
α -Bisabolol	0.21	0.15
Sesquisabinene hydrate	0.30	0.29
Zingiberenol	0.15	0.13
Guaicol	0.22	0.14
Zingerone	7.49	3.42
β -Eudesmol	0.21	0.11
Sesquiterpene alcohol	0.64	0.30
Phenyl curcumene	0.05	0.14
6-Paradol	0.50	0.17
6-Shogaol	6.30	2.35
6-Ginger dione	1.92	1.00

Source: Bartley and Jacons (2000).

Qi Han *et al.* (1998) found variation in oil content and menthol content in micropropagated mint plants compared to control. Some somaclones exceeded controls in oil and menthol contents by 27.77% and 8.16–10.86%, respectively. Kumar and Bhatt (1999) found mint oil effective as a bioinsecticide against *Amritodus atkinsoni* and *Scirtothrips mangiferae*. Saxena and Singh (1998) studied the effects of irrigation, mulch and nitrogen on yield and composition of Japanese mint (*Mentha arvensis* subsp. *haplocalyx* var. *piperascens*) oil. They found essential oil from the first harvest was richer in menthol (78.8%) than the oil obtained from second harvest (75.2% menthol).

Croteau (1991) reviewed metabolism of monoterpenes in mint (*Mentha*) species. The biosynthesis and catabolism of C3- and C6-oxygenated p-menthane monoterpenes, cyclization of geranyl pyrophosphate to their precursor (–)-limonene, the metabolism of limonene, the developmental regulation of monoterpene metabolism and its potential role in the defence mechanisms of *Mentha* species are discussed. Monoterpene biosynthesis tends to occur mainly in young leaves; whereas catabolic activities increase at maturity, in parallel with oil gland senescence. It is concluded that for commercial mint oil production a dynamic balance between biosynthetic and catabolic processes is essential.

Spencer *et al.* (1990) evaluated the production of terpenes by differentiated shoot cultures of *Mentha citrata* transformed with *Agrobacterium tumefaciens* T37. The shoot cultures synthesized a mint oil fraction which contained the major terpenes characteristic of the parent plant in quantities similar to those in intact tissue. Oil glands were observed to be present on the leaves of the transformed culture. In the mint condensate they were 1-menthol, menthone and neomenthol (Machale *et al.* 1997).

Essential oil glandular trichomes are the specialized anatomical and structural characteristic of plants accumulating significant quantities of commercially and pharmaceutically valuable essential oil terpenoids. The developmental dynamics of these structures together with the oil secretory process and mechanisms have a direct bearing on the secondary metabolite production, sequestration, and holding potential of the producer systems. The essential oil gland trichomes of menthol mint leaf have been stereologically analyzed to discern their anatomical archetype *vis-à-vis* volatile oil secretion and sequestration as integrated in the overall leaf ontogeny. Cuticular 'dehiscence' or decapping, leading to collapsing of the peltate trichomes was a notable characteristic of the menthol mint oil glands. Ecophysiological, evolutionary, phytopharming and biotechnological connotations of the novel phenomenon have been hypothesized (Sharma *et al.* 2003).

Ozel and Ozguven (2002) conducted field experiments to determine the effect of different planting dates on the essential oil components of different mint varieties (*Mentha arvensis* var. *piperascens*, *M. piperita* cultivars Mitcham, Eskisehir, and Prilubskaja). The mint oil components, i.e., α -pinene (0.49–1.00%), β -pinene (1.38–2.12%), 1,8-cineole (eucalyptol) (2.64–10.85%), menthone, menthofuran (28.09–49.52%), menthol (22.55–38.89%), pulegone (0.00–1.32%), menthyl acetate (0.46–6.78%), and β -caryophyllene (0.54–2.84%), were determined. The results indicated that the essential oil components were affected by planting date, mint cultivar, and cutting numbers. The highest menthol ratio was obtained from *M. arvensis* var. *piperascens* (33.50–38.89%) from second cutting and autumn transplanting. Frerot *et al.* (2002) reported a new p-menthane lactone from *Mentha piperita* L 3,6-dimethyl-4,5,6,7-tetrahydro-benzo(b)-furan-2(3H)-one (Menthofuroolactone)

Nutmeg

Dried nutmeg and mace are used as spices and also for extracting oil and oleoresins. Mallavarapu and Ramesh (1998) indicated the nutmeg oil composition as follows: α -thujene (2.2%), α -pinene (13.6%), camphene (0.3%), sabinene (32.1%), β -pinene (12.9%), myrcene (2.2%), δ -3-carene (0.8%), α -phellandrene (0.7%), α -terpinene (2.2%), p-cymene (0.7%), limonene (4.0%), 1,8-cineole + β -phellandrene (2.3%), γ -terpinene (3.9%), trans-sabinene hydrate (0.5%), terpinolene (1.2%), linalool (0.8%), cis-p-menth-2-en-1-ol (0.4%), trans-p-menth-2-en-1-ol (0.3%), terpinen-4-ol (7.2%), α -terpineol (0.8%), safrole (2.8%), eugenol (0.4%), methyl eugenol (1.6%), β -cubebene (0.1%), β -caryophyllene (0.2%), trans- α -bergamotene (0.1%), (E)-methyl isoeugenol (0.2%), germacrene D (0.1%), myristicin (2.6%) and elemicin (2.4%).

Lawrence (2000) compared the oil composition from various sources such as the West Indian nutmeg oils, fresh and dried nutmeg pericarp oil and mace oil using different GC stationary phases. Gopalakrishnan (1992) studied the chemical composition of nutmeg and mace oil. β -pinene and sabinene dominated in both the oils (Table 11.16). Maya *et al.* (2004) reported myristicin as high as 45% in Indian nutmeg oil and 36.6% in Indian mace oil. Mallavarapu and Ramesh (1998) reported nutmeg oil having 76.8% monoterpenes, 12.1% oxygenated monoterpenes and 9.8% phenyl propanoid ether. They also reported mace oil with 51.2% monoterpenes, 30.3% oxygenated monoterpenes and 18.8% phenyl propanoid ether. Their study indicated that in quality, Indian nutmeg oils are intermediate between East Indian and West Indian oils.

Ehlers *et al.* (1998) using HPLC analyzed nutmeg and mace oils produced by supercritical CO₂ extraction and compared it with steam distilled oils and also with oils of East Indian, West Indian and Papuan origin. Myristicin in nutmeg oil of East Indies ranged from 17.5–25.9% and West Indies 2.8–3.7%. Mace oil of whole blades from East Indies contain myristicin 19.1–24.6%, West Indies 4.4–9.1% and that of Papua 1.1–1.4%. Oil yield from raw material was high in the supercritical extraction. Myristicin, the hallucinogenic principle of nutmeg oil, was high in the steam distilled oil. Safrole content in the nutmeg and mace oil of the East Indies ranged from 2.5–3.7% while safrole was very high in the mace oil from Papua (20.5–30.7%). Elemicin was high in the West Indies (3.9–10.1%) and Papua oils (2.1–3.0) compared to East Indian oil (nutmeg: 0.5–1.5%, mace 0.4–0.7%).

Rosemary

Rosemarium officinalis is an aromatic plant, widely used in the pharmaceutical, perfumery and food industries. Steam distillation of the fresh leaves and flowering tops yield 1–2% oil (Boutekedjiret *et al.* 1997). The main constituents of rosemary oil are α -pinene, camphor, cineole, borneol and bornyl acetate. Wide variability occurs in the chemical composition of rosemary oil of different countries (Arnold *et al.* 1997, Dellacassa *et al.* 1999, Fournier *et al.* 1989, Lawrence 1995). Mainly there are two types of rosemary oil in trade, Tunisian and Moroccan, having 1,8 cineole (38–55%) and Spanish with camphor (12.5–22.0%) and cineole (17–25%) (Arnold *et al.* 1997, Mallavarapu 2000). The leaves of rosemary grown in the Kumaon hills of Uttaranchal contained 0.25–0.52% volatile oil on fresh weight basis (Kumar *et al.* 2004). The chief components of oil were α -pinene (14.90%), 1,8-cineole (17.50%), camphor (12.7%), borneol (5.50%) and verbenone (11.00%) (Table 11.17).

Studies conducted to determine the effect of different temperatures during the drying process on the amount and quality of essential oils of rosemary (*Rosmarinus*

Table 11.16 Composition of nutmeg and mace oil

Compound	Composition	
	Nutmeg oil	Mace oil
α -Pinene	14.72	15.24
β -Pinene + Sabinene	62.66	45.52
α -Phellandrene	3.06	3.17
Δ^3 -Carene	0.60	0.67
α -Terpinene + P-Cymene	1.08	3.53
1,8-Cineole + Limonene	6.18	6.97
β -Phellandrene	1.08	2.80
γ -Terpinene	0.54	1.83
Linalool + Terpinolene	0.48	0.42
β -Terpineol	0.25	0.32
Borneol (tentative)	0.05	0.16
Terpinen-4-ol	1.85	4.59
α -Terpineol + Piperitol	0.36	0.94
Geraniol	0.02	0.22
Safrole + p-Cymene-8-ol	0.53	0.67
Bornyl acetate	0.07	0.09
Methyl eugenol	0.14	0.22
Eugenol + terpenyl acetate	0.22	0.15
Geranyl acetate + α -Copaene	0.29	0.16
Isoeugenol (<i>cis</i>)	0.31	0.45
β -Caryophyllene + isoeugenol (<i>trans</i>)	0.07	0.07
α -Humulene	0.02	0.03
δ -Cadinene	0.08	0.15
Myristicin	3.28	5.92
Elemicin	1.38	3.14
Myristic acid	0.01	0.01
Trimyristin	0.06	0.05

Source: Gopalakrishnan (1992).

officinalis) indicated that higher drying temperature decreased the essential oil content (% v/w) from 2.13 (40 °C) to 1.62 (60 °C) and 1.09% (80 °C). Essential oil composition was similar, except for camphor at 40 °C and 60 °C. However, concentrations of alpha-pinene, beta-myrcene and camphor were decreased at 40 °C and 80 °C (Blanco *et al.* 2002).

Tucker and Maciarello (1986) reported α -pinene, camphene, 1,8-cineole, camphor, bornyl acetate and borneol as the major compounds in five varieties of rosemary oil. Rosemary oil from Argentina contained 20 components of which the major ones were α -pinene, myrcene, 1,8-cineole, camphor and β -caryophyllene (Mizaahi *et al.* 1991). Lawrence (1995) reported that rosemary oil from Spain and Portugal contained 30–50% oxygenated monoterpenes where as the oils of Moroccan, Tunisian and Yugoslavian origin contained 70–80% oxygenated monoterpenes.

Rao *et al.* (1998) compared the oil loss in rosemary leaves by convection and microwave drying methods. The loss of volatile oil was less (7.25%) during convection drying while microwave drying led to a loss of 61.45%. The volatile oil of fresh rosemary contained mostly monoterpenes and their derivatives (95–98%). The major components of rosemary leaf oil were camphor (23.9–34.0%) and 1,8-cineole (15.5–29.8%).

Boutekedjiret *et al.* (2003) reported that oil yield from the hydrodistilled herbage

Table 11.17 Volatiles from rosemary

Compound	% Composition
α -Pinene	5.5–26.0
Camphene	1.5–13.0
β -Pinene	2.60
Myrcene	1.50
Δ -3-Carene	2.30
Limonene	2.80
p-Cymene	1.80
1,8-Cineole	9.4–55.0
γ -Terpinene	1.30
Terpinolene	0.50
Linalool	0.5–4.9
Camphor	5.0–26.4
Isoborneol	0.30
Borneol	1.1–5.5
Dihydrocarveol	0.13
Verbenone	0.0–14.1
Linalyl acetate	1.40
Bornyl acetate	1.90
β -Caryophyllene	1.40
α -Humulene	1.20
Methyl isoeugenol	0.50
<i>Trans</i> - β -farnesene	0.40
γ -Muuroolene	0.30
δ -Cadinene	1.20
β -Sesquiphyllylandrene	1.30
Carophyllene oxide	0.60
Humulene epoxide	0.06
α -Bisabolol	0.78
Unidentified	1.93

Source: Kumar *et al.* (2004).

was lower (0.44%) than that of steam distilled herbage (1.2%). The steam distilled oil contained 52.4% 1,8-cineole where as the hydrodistilled oil contained much less (31.9%) cineole. The contents of camphor (19.7%), borneol (12.1%) and α -terpineol (12.8%) were higher in hydrodistilled oil compared to the steam distilled oil.

Spearmint

Tsuneya *et al.* (1998) studied acidic components in Scotch spearmint oil (*Mentha gracilis* Sole) and 46 acidic components (including 35 carboxylic acids and 11 phenols) were identified. Three carboxylic acids peculiar to *M. gracilis* were identified from spectral data: *cis*-2-pentylcyclopropane-1-carboxylic acid, 3-isopropenylpentane-1,5-dioic acid and 3-isopropenyl-6-oxoheptanoic acid.

Platin *et al.* (1994) studied equilibrium distributions of key components of spearmint oil in sub/supercritical carbon dioxide. Effects of temperature (at 35 °C, 45 °C or 55 °C) and pressure (10–110 atm) on the relative distribution coefficients of 12 key components (6 monoterpenes, 3 monoterpenoids and 3 sesquiterpenes) of spearmint oil (essential oil of *Mentha cardiaca* (*M. gracilis*); Scotch spearmint) at equilibrium in dense CO₂ were investigated under conditions ranging from subcritical to supercritical regions. At 35 °C all key components of spearmint oil were equally soluble in dense CO₂ within the 12–102 atm pressure region. Vapour-pressure effects, coupled with

the decrease in solvating power, dominated the effects of polarity and molecular mass of the key components. The quality of essential oils decreased with increasing fraction of monoterpenes, and it is concluded that deterpenation of spearmint oil with dense CO₂ is possible either at 45 °C/27 atm or 55 °C/35 atm, where the monoterpene hydrocarbons tend to concentrate, and can be preferentially recovered.

Ishihara *et al.* (1992) reported new pyridine derivatives and basic components in spearmint oil (*Mentha gentilis* f. *cardiaca*) and peppermint oil (*Mentha piperita*). A total of 38 nitrogen-containing components including 11 new pyridine derivatives, 2-isopropyl-4-methylpyridine, 4-isopropenyl-2-methylpyridine, 2-ethyl-4-isopropenylpyridine, 2-acetyl-4-isopropylpyridine, 2,4-diisopropenylpyridine, 2-acetyl-4-isopropenylpyridine, 4-acetyl-2-isopropenylpyridine, 5-[(Z)-1-buten-1-yl]-2-propylpyridine, 5-[(E)-1-buten-1-yl]-2-propylpyridine, 3-[(Z)-1-buten-1-yl]-4-propylpyridine and 3-[(E)-1-buten-1-yl]-4-propylpyridine, were identified by comparing their spectroscopic data with those of synthetic samples. Among them, 2-acetyl-4-isopropenylpyridine was a major component with a powerful grassy-sweet and minty odour.

Ringer *et al.* (2005) made a detailed review on monoterpene metabolism, cloning, expression and characterization of (–)-isopiperitenol/(–)-carveol dehydrogenase of peppermint and spearmint. They stated that the isolation of the genes specifying redox enzymes of monoterpene biosynthesis in mint indicates that these genes arose from different ancestors and not by simple duplication and differentiation of a common progenitor, as might have been anticipated based on the common reaction chemistry and structural similarity of the substrate monoterpenes. The full-length spearmint dehydrogenase shares >99% amino acid identity with its peppermint homolog and both dehydrogenases are capable of utilizing (–)-*trans*-isopiperitenol and (–)-*trans*-carveol. These isopiperitenol/carveol dehydrogenases are members of the short-chain dehydrogenase/reductase superfamily and are related to other plant short-chain dehydrogenases/reductases involved in secondary metabolism (lignan biosynthesis), stress responses, and phytosteroid biosynthesis, but they are quite dissimilar (approximately 13% identity) to the monoterpene reductases of mint involved in (–)-menthol biosynthesis.

The undesirable top notes or off-notes found in mint, clary sage, and cedarwood oils could be quantitatively determined using a non-equilibrated solid phase microextraction/gas chromatography/selected ion monitoring/mass spectrometry (SPME/GC/SIM-MS) technique. Using the low threshold components, dimethyl sulfide, 2-methylpropanal, 2-methylbutanal, and 3-methylbutanal, which are associated with the off-notes of these oils, their levels could be quantitatively determined. The highest level of off-notes was found in a sample of Scotch spearmint oil where the levels of the four constituents were, dimethyl sulfide (238 $\mu\text{g g}^{-1}$), 2-methylpropanal (286 $\mu\text{g g}^{-1}$), 2-methylbutanal (1048 $\mu\text{g g}^{-1}$) and 3-methylbutanal (1489 $\mu\text{g g}^{-1}$). These quantitative results in combination with sensory evaluations could provide for a powerful overall assessment of essential oil quality (Coleman *et al.* 2004).

A study was conducted to identify the fragrance compounds of *Mentha spicata* oil from Cameroon and its solid-phase microextraction (SPME)-headspace by means of gas chromatograph spectroscopy (GC and GC-MS) and olfactory methods (GC-sniffing technique and olfactory correlations) to determine the importance of each single constituent with their specific odour attributes. The odour impression was very pleasant in spearmint, with green, floral, fruity, and spicy side notes. The composition of the spearmint essential oil and its corresponding SPME-headspace sample was

very similar and differed only in the concentrations of the main compounds, namely, (–)-limonene (essential oil: 6.55%, SPME: 8.31%), 1,8-cineole (4.19%, 7.12%) and *trans*-1-hexen-3-ol (0.66%, 1.72%). In addition to the composition of both samples, the olfactory evaluations certify a high quality of the essential oil and its possible use in food, perfumery, and cosmetic products requiring a fresh-spearmint odour (Jirovetz *et al.* 2002).

The major chemical constituents of the hydrodistilled essential oil and their major isolates from cultivated *M. spicata* was identified by IR, ¹H- and ¹³C-NMR and GC. (S)-(–)-limonene (27.3%) and (S)-(–)-carvone (56.6%) (representing 83.9% of the spearmint oil) and (R)-(+)-limonene (21.4%), dihydrocarvone (5.0%), (R)-(+)-carvone (50.4%) and dillapiole (17.7%), respectively. *In vitro* biological activity evaluation of the isolated oil components revealed that both the optical isomers of carvone were active against a wide spectrum of human pathogenic fungi and bacteria tested. (R)-(+)-Limonene showed comparable bioactivity profile over the (S)-(–)-isomer. The activity of these monoterpene enantiomers was found to be comparable to the bioactivity of the oils in which they occurred (Aggarwal *et al.* 2002).

Thyme

The volatile oil of Egyptian *T. vulgaris* was richer in linalool and terpene hydrocarbons. The oil contained thymol and carvacrol in only moderate concentrations. The highest thymol and carvacrol concentrations were observed during the beginning of flowering (Karawya and Hifnawy 1974). Commercial samples of Ethiopian thyme (*T. schimperi*) contained carvacrol and thymol (Lemordant 1986).

Oszagyan *et al.* (1996) compared the composition of steam distilled and SFE oils. SFE product contained 10–15% thymol and 30–35% carvacrol while steam distilled oil contained 48–50% thymol and 8–10% carvacrol. Cuban thyme oil contained thymol (34.6%), γ -terpinene (17.61%) and p-cymene (17.65%) as major components (Pino *et al.* 1997). Fresh plant material from Bulgarian thyme (*T. vulgaris*) yielded 0.46% essential oil (Stoeva *et al.* 2001).

Studies on the effect of harvest time on yield and oil composition of thyme (*T. mongolicus*) indicated that the best time of harvest for the highest oil yield and high thymol and carvacrol content was during or immediately after the full bloom (Fan-ming and Chen-Jin 2002). Asllani and Toska (2003) evaluated Albanian thyme oils, which were dominated by p-cymene (7.76–43.75%), γ -terpinene (4.20–27.62%), thymol (21.38–60.15%) carvacrol (1.15–3.04%) and β -caryophyllene (1.30–3.07). Thyme (*T. pulegioides*) growing wild in Lithuania contained five chemotypes (i) linalool type, (ii) geranial/geraniol/neral type, (iii) thymol type, (iv) carvacrol/ γ -terpinene/p-cymene type and (v) thymol/carvacrol/p-cyme/ γ -terpinene type (Loziene *et al.* 2003).

The constituents of essential oils isolated by hydrodistillation of aerial parts of *Satureja hortensis*, used as thyme in Turkey recorded α -terpinene (2.34 and 2.66%), p-cymene (21.82 and 14.64%), γ -terpinene (18.92 and 23.09%) and β -caryophyllene (3.75 and 4.56%), as the main components (Ozcan and Chalchat 2004). Commercial essential oils of thyme from different geographical areas of Italy and France were rich in thymol (22–38%) and its biogenetic precursors, namely, γ -terpinene and p-cymene (Zambonelli *et al.* 2004). The main constituents of the hydro-distilled essential oil from the herb of lemon thyme (*Thymus citriodorus* L.) cultivated in Iran were geraniol (54.4%), geranial (13.9%), neral (10.1%), nerol (5.2%), 3-octanone (3.3%) and borneol (3.2%) (Omidbaigi *et al.* 2005).

Turmeric

Volatile oils are extracted from rhizomes and leaves of turmeric. The chemical composition of volatiles from various parts of turmeric has been investigated extensively. The oil yield and composition show wide variation depending on geographic conditions, variety, agronomic practices, maturity at harvest and post-harvest processing. GC-MS analysis of the oil indicated the presence of as many as 84 components in the oil in varying levels. Volatile oil content in turmeric rhizomes ranged from 1.3–5.5% (Guenther 1961b). The chief constituents of rhizome oil were turmerone, ar-turmerone and turmerol (Govindarajan 1980). The rhizome oil contained limonene, cineole, curcumene, zingiberene, bisabolene, β -phellandrene, ar-turmerone and turmerone (Gopalani and Ratnambal, 1987). The rhizome oil of Indonesian origin was constituted by the following compounds: ar-turmerone (41.4%), turmerone (29.5%), turmerol (10%) and α -atlantone (2.4%) (Zwaving and Bos, 1992).

Nigam and Ahmad (1991) reported 59.7% ar-turmerone in the rhizome oil. The oil from Malaysian rhizomes was dominated by α -turmerone (45.3%), linalool (14.9%) and β -turmerone (13.5%) (Ibrahim *et al.* 1999). Among six turmeric cultivars grown in Maharashtra namely, Rajapuri, Krishna, Mydukur, Salem, Tekurpetta and Armoor, the highest essential oil contents were recorded in mother rhizomes of Mydukur and fingers of Salem (Rakhunde *et al.* 1998). Garg *et al.* (1999) reported that oil content in the rhizomes of 27 accessions from North Indian Plains varied between 0.16% and 1.94% on fresh weight basis. Based on the contents of β -pinene, p-cymene, α -curcumene, β -curcumene, ar-turmerone, α -turmerone and β -turmerone the accessions were classified into two groups: (i) those in which the sum of the seven major terpenes was in the range 58–79%, (ii) those in which the sum was 10–22%. The rhizome oil from Bhutan was constituted by 30–32% α -turmerone, 17–26% ar-turmerone and 15–18% β -turmerone (Sharma *et al.* 1997).

Gopalan *et al.* (2000) noticed that during supercritical carbon dioxide extraction, the solubility of turmeric oil was maximum at 313–333 K and 20–40 MPa and about 60% of the oil was composed of turmerone and ar-turmerone. Fresh rhizome oil from Pakistan was abundant in ar-turmerone (31.1–41.2%) and turmerone (9–11.1%) (Riaz *et al.* 2000). Iron deficiency significantly increased the essential oil and curcumin contents in turmeric rhizomes (Dixit *et al.* 1999).

Chatterjee *et al.* (2000) reported that no detectable differences were observed in the aroma impact compounds of γ -irradiated and commercial volatile oils. The rhizome oil of *C. longa* cv. Roma from North Indian Plains was rich in 1,8-cineole (11.2%), α -turmerone (11.1%), β -caryophyllene (9.8%), ar-turmerone (7.3%) and β -sesquiphellandrene (7.1%) (Raina *et al.* 2002). The rhizome essential oils of *C. longa* cv Roma grown in Indo-Gangetic plains were rich in α - and β -turmerones (40.8%), myrcene (12.6%), 1,8-cineole (7.7%) and p-cymene (3.8%) (Bansal *et al.* 2002). The turmeric oils from Calicut (South India) was dominated by ar-turmerone (31.1%), turmerone (10.0%), curlone (10.6%), ar-curcumene (6.3%), p-cymene (3.0%), β -sesquiphellandrene (2.6%), β -phellandrene (2.4%) and dehydrocurcumene (2.2%). The root oil also contained ar-turmerone (46.8%) as the chief component followed by ar-curcumene (7.0%), dehydrocurcumene (4.3%) and p-cymene (3.3%) (Leela *et al.* 2002). The rhizomes from Reunion Island yielded 1.1% oil, which contained ar-turmerone (21.4%), terpinolene (15.8%), zingiberene (11.8%), ar-turmerol (7.7%), β -turmerone (7.1%), sesquiphellandrene (8.8%) and β -caryophyllene (5.7%) as major compounds (Chane-Ming *et al.* 2002) (Table 11.18).

The essential oil from Cuban rhizomes was reported to contain 47.7% ar-turmerone

Table 11.18 Chemical composition (%) of essential oils of rhizome, leaves and flowers of *Curcuma longa* L. from Reunion Island (HP-5 column)

Compounds	Rhizomes	Leaves	Flowers
Tricyclene	–	0.1	0.1
α -Pinene	0.2	0.7	–
α -Fenchene	–	0.1	0.8
Sabinene	–	0.1	0.1
β -Pinene	–	0.7	0.6
Myrcene	0.3	1.4	2.1
δ -2-Carene	–	0.1	0.2
α -Phellandrene	1.0	2.8	3.6
δ -3-Carene	0.3	1.2	1.7
α -Terpinene	1.4	3.7	4.4
p-Cymene	0.6	0.3	0.4
Limonene	–	–	0.4
1,8-Cineole	2.0	4.6	4.6
(Z)- β -Ocimene	–	0.4	0.8
(E)- β -Ocimene	–	0.7	1.8
γ -Terpinene	–	0.4	0.8
p-Cymenene	0.4	–	–
Terpinolene	15.8	76.8	67.4
Linalool	–	0.7	0.5
p-Mentha-1,3,8-triene	–	0.2	0.3
p-Cymen-7-ol	–	–	0.2
Terpinen-4-ol	0.2	–	–
p-Cymen-8-ol	–	0.2	0.3
α -Terpineol	–	0.3	0.3
2-Undecanone	–	–	0.2
Geranyl acetate	–	–	0.1
<i>Cis</i> - α -bergamotene	0.3	–	–
β -Caryophyllene	5.7	0.1	0.2
α -Humulene	1.4	–	–
(E)- β -Farnesene	0.6	0.1	0.1
ar-Curcumene	4.5	0.1	0.1
Zingiberene	11.8	1.0	1.3
β -Bisabolene	1.9	0.1	0.2
β -Sesquiphellandrene	8.8	0.4	0.5
(E)- γ -Bisabolene	0.7	–	–
(E)-Nerolidol	0.2	0.1	0.2
ar-Turmerol	0.3	–	–
ar-Dehydro-turmerone	0.6	–	–
ar-Turmerone	7.7	–	–
α -Turmerone	21.4	–	–
β -Turmerone	7.1	–	–
(Z)- γ -Atlantone	–	0.1	0.9
Germacrone	–	0.1	0.1
Curcuphenol	0.2	–	–

Source: Chane-Ming *et al.* (2002).

and 16.1% turmerone as major compounds (Pino *et al.* 2003). Turmeric rhizomes from Gorakhpur region (North India) was reported to contain 1.6% oil and ar-turmerone, ar-turmerol, β -bisabolene and zingiberene as chief components (Singh *et al.* 2003). Rhizome oil extracted by the solid phase microextraction method contained ar-curcumene, ar-turmerone, zingiberene, β -sesquiphellandrene, sabinene, 1,8-cineole and 1,4-terpineol as major components (Mata *et al.* 2004). The rhizome oil of *Curcuma*

longa from the lower Himalayan region was rich in α -turmerone (44.1%), β -turmerone (18.5%) and ar-turmerone (5.4%) (Raina *et al.* 2005).

The leaves of turmeric yield 0.37–2.5% volatile oil. The leaf oil from Nigeria contained mainly monoterpenes with 47.7% α -phellandrene and 28.9% terpinolene (Oguntimein *et al.* 1990). The leaf oil from Kerala (South India) was dominated by 56.7% α -phellandrene and 11.8% terpinolene (McCarron *et al.* 1995). The leaf oil of Vietnam origin contained 2.5% oil (dry weight basis) and was dominated by the monoterpenes, α -phellandrene (24.5%), 1,8-cineole (15.9%), p-cymene (13.2%) and β -pinene (8.9%) (Dung *et al.* 1995). The leaf oil from Bhutan was dominated by α -phellandrene (18.2%), 1,8-cineole (14.6%) and p-cymene (13.3%) (Sharma *et al.* 1997). The turmeric leaves from South India yielded 1.3% volatile oil. The oil was dominated by α -phellandrene (32.6%), terpinolene (26.0%), 1,8-cineole (6.5%) and p-cymene (5.9%) (Leela *et al.* 2002). The leaf petiole and lamina oils of *C. longa* cv. Roma were rich in myrcene (35.9%), 1,8-cineole (12.1%) and p-cymene (12.7%) (Bansal *et al.* 2002). *C. longa* leaf oil from North Indian Plains was mainly constituted by p-cymene (25.4%), 1,8 cineole (18%), *cis*-sabinol (7.4%) and β -pinene (6.3%) (Garg *et al.* 2002).

The leaf oil of *C. longa* cv. Roma contained terpinolene (26.4%), 1,8-cineole (9.5%), α -phellandrene (8%) and terpinene-4-ol (7.4%) as chief constituents (Raina *et al.* 2002). The leaf oil of *C. longa* var Rasmi from Orissa was reported to contain α -phellandrene (38.24%), C-8 aldehyde (20.58%), 1,8-cineole (8.64%), α -pinene (2.88%) and β -pinene (2.36%) as chief constituents (Behura *et al.* 2002). The fresh leaves of Bhutan origin contained 0.37% to 0.42% oil and the main constituents were α -phellandrene (18.2%), 1,8-cineole (14.6%) and p-cymene (13.3%) and terpinolene (11.6%) (Sharma *et al.* 1997). The leaves of turmeric from Reunion Island yielded 0.5% volatile oil. The major constituent in the leaf oil was terpinolene and it differs from the oils of other origins in its high level of terpinolene (76.8%) and its small amount of phellandrene (2.8%) (Chane-Ming *et al.* 2002) (Table 11.18). The leaf oil of turmeric from the lower Himalayan region contained α -phellandrene (53.4%), terpinolene (11.5%) and 1,8-cineole (10.5%) as major constituents (Raina *et al.* 2005).

Freshly harvested flowers of turmeric from South India yielded 0.3% volatile oil. Twenty-five components contributing to 52% of the oil were identified among which p-cymen-8-ol (26%), terpinolene (7.4%) and 1,8-cineole (4.1%) were the major components (Leela *et al.* 2002). The flowers of *C. longa* from Reunion island contained 0.1% volatile oil and the oil was dominated by terpinolene (67.4%), 1,8-cineole (4.6%), α -terpinene (4.4%), α -phellandrene (3.6%) and myrcene (2.1%) (Chane-Ming *et al.* 2002) (Table 11.18).

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