CHAPTER II

BACKGROUND

1. Spice Oleoresins

A closer approximation to the total spice flavor is given by its oleoresin. An oleoresin is an extremely concentrated, viscous, resinous extract obtained by solvent extraction and containing all the flavoring ingredients of spice soluble in the particular organic volatile solvent used. The oleoresins are prepared from the ground dry spice by extraction with a suitable volatile organic solvent or a mixture of solvents; unless great care is taken about the purity of such solvents, traces of the higher boiling fractions may remain in the oleoresin during stripping of the solvent and give it a decided off-aroma. On the other hand, excessive vacuum and heat treatment to remove last traces of solvent, now specifically required by federal regulations of different countries, invariably results in a degree of damage to the heat-labile flavor components or, in extreme cases, to an almost complete loss of the top notes of the essential oil present.

An oleoresin plant or oleoresin unit consists of an extractor for solvent extraction of spices, desolventization units for meal and miscella, and blending units, Jacketed vessles are used for desolventization. Removal of the last traces of solvent poses problems, owing to high viscosity and heat sensitivity. Scraped film evaporators are somtimes employed for this purpose. Handling of resins for blending is also a problem, owing to high viscosity and pungency.

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Solvent recovery and yield of resin dictate the economics of these units.

As the name implies, oleoresin consists of a blend of the essential oil and resinous matter of the spice and related compounds, nonvolatile fatty oil, and coloring matter soluble in the particular solvent used. The importance of choice of solvent can best be illustrated by considering the extraction of a typical spice-say, turmeric. If one extracts turmeric with petroleum ether, the product is a light, fluid oleoresin, which is highly aromatic and smells strongly of ground turmeric, but has little of its yellow coloring power. If, however, one extracts it with acetone, one gets a brilliant yellow extractive, which is a hard solid with only a very small amount of the characteristic odor. Both of these products are strictly oleoresins, but the difference in their physical condition, chemical composition, aroma, and coloring power is determined entirely by the nature of the solvent that is used in their preparation. Since the herbs and spices are variable, it follows that the aromatic extractives obtained from them will also be variable, both in flavor characteristics and in flavor strength.

2. The advantages and disadvantages of spice oleoresins, according to Health (1972), are as follows :

A. Advantages of Oleoresins

1. They are hygienic, being free from bacteria, etc.
2. They can be standardized for flavoring strength.
3. They contain natural antioxidants.
4. They are free from enzymes.
5. They have a long shelf-life under ideal conditions.
6. They have less bulk in storage.
7. They have less weight in shipping.
8. There is no color disturbance or specks.

9. There is no danger of molding as in spices.

B. Disadvantages of Oleoresins

1. The flavor is good but as variable as the raw material.

2. They are very concentrated and hence difficult to handle and weight accurately.

3. They range from liquids to viscous solids, which are difficult to incorporate into food mixes without hot spots.

4. Tannins are present unless they are specially treated and freed.

5. Flavor quality depends on the solvent used and also on the raw material.

3. Factor Affecting Yield and Quality of Oleoresins

The following factors significantly affect both the yield and the quality of oleoresin.

3.1 Selection of Proper Variety of Spice

Distinct differences in quality and yield of both essential oils and oleoresins have been noticed in varieties of important spices such as cardamom, chilies, ginger, pepper, and turmeric grown in India (Lewis, 1972). There are four important commercial varieties of ginger in the world market, which differ in appearance, texture, yield, and quality of oil and oleoresin or nonvolatile extract (Table 1). The oleoresin content of pepper varies from 10 to 12%, chilies 12 to 16%, ginger 5 to 7%, and turmeric 4 to 7% (Lewis *et al.*, 1974). In twenty-seven varieties of ginger grown in India, the nonvolatile extractives obtained by the cold percolation technique varied from 1.05 to 3.95% when ethylene dichloride was used as solvent, and from 1.9 to 8.34% when alcohol was the solvent (Lewis *et al.*, 1973). Likewise, Cowley (1972) has shown differences in yield and quality of oleoresin from vanilla beans grown in different regions of the world. Suzuki *et al.* (1957) and Mathew *et al.* (1971a,b) have also shown wide variations in oleoresin content of several commerical varieties of chilies grown in different regions of the world.

Table 1 Volatile oil and oleoresin content of internationally important commercial varieties of ginger [a]

Variety	Appearance	Flavor characteristics	Volatile oil (% v/w)	Nonvolatile extract (EDC)[b] (%)
Cochin	Bold, light-brown partly peeled	Lemon-like odor and flavor	2.2	4.25
Jamaican	Bold, very light buff color, clearly peeled	Delicate aroma and flavor	1.0	4.4
Sierra Leone	Plump, dark, partly peeled from sides	Pungent and slightly camphoraceous flavor	1.6	7.2
Nigerian	Bold, light color, partly peelped fibrous	Very pungent, camphoraceous flavor	2.5	6.5
Japanese [c]	Dark, bold, unpeeled	-	0.5	4.6

[a] From Lewis *et al.* (1972).

[b] Ethylene dichloride.

[c] Commercially not very important.

3.2 Conditioning or Drying Spices

The spice selected must be dried thoroughly to bring down the moisture to a critical level, which is different from the critical moisture content for the prevention of mold growth. This level also depends on the nature of the solvent to be used. For water-soluble solvents such as alcohol and acetone, the moisture level should be very low; otherwise it dilutes the solvent, and this, in turn, lowers the solubility of certain flavoring compounds. Besides, if spices are not dried, they require larger quantities of solvent and larger equipment, with the added difficulty of extraction mentioned above.

3.3 Preparation of Material for Pulverizing

In chilies, the calyx should be removed so that it does not imprat a greenish tinge to the oleoresin. In ginger and turmeric, the hands and fingers should be coarsely cracked into small pieces before they are fed to the pulverizer. Properly dried spices should be cleaned to get rid of all extraneous matter. They are then pulverized to 30- to 40-mesh size. Although fine grinding of spices may give slightly higher yields, it has the disadvantage of caking and channeling, as in ginger and pepper. Chili powders of 0.5- and 0.35-mm mean mesh gave yields of 12% and 16% oleoresin, respectively (Nambudiri *et al.*, 1970; Mathew *et al.*, 1971a,b).

3.4 Choice of Solvent

The selection of solvent has received due attention from several workers. Sabel and Warren (1972) reported that, out of a number of solvents studied, acetone, ethanol, and isopropanol could be used very satisfactorily for the recovery of oleoresin from ginger. There was little to chose among these three solvents. Mathew *et al.* (1971a) studied the comparative efficiency of hexane, ethanol, and ethylene dichloride and concluded that hexane was a poor solvent for extraction of capsaicin from capsicums or chilies. On the other hand, alcohol did not extract the color efficiently. Furthermore, alcohol, especially hot alcohol, gives a product that is semisolid instead of the usual free-flowing products. Thus, ethylene dichloride is a good solvent for the extraction of chili oleoresin, getting yields up to 13 to 14%.

Acetone and alcohol have been widely used for ginger extraction. Of course, acetone is costlier and more volatile than alcohol. However, the quality of oleoresin from acetone was found to be better than that from alcohol. In some varieties of ginger, the yield of oleoresin was too high (up to 20%) (Connel, 1970a,b; Lewis *et al.*, 1972). Because of the dilution of the solvent with the water present in the spice and hence the chances of extraction of more nonflavor components, it is preferable to use a water-immiscible volatile organic solvent such as ethylene dichloride for extraction. Lewis *et al.* (1972, 1974) have given the comparative values for four solvents (acetone, alcohol, hexane, and ethylene dichloride) with the cold percolation method of extraction.

3.5 Conditions of Extraction

Several conditions of extraction, such as the time taken for the solvent to wet the bed of material and the rate of flow of the extract, are commonly influenced by the size of the particles, the density of packing, the bed height, the area, the rate of percolation, etc. Optimum conditions for quick and efficient extraction have thus to be worked out for each spice, since time is a very important factor in commercial operation of the process. The method of extraction is yet another important factor to consider. Systematic work done at CFTRI, Mysore, shows that three-stage batch countercurrent extraction is satisfactory for all the major spices (pepper, ginger, chilies, turmeric, etc.). In addition, ethylene dichloride has been found to be the most suitable solvent because (1) the final product is clear and (2) open distillation can be used for desolventization (Nambudiri *et al.*, 1970; Mathew *et al.*, 1971a; Lewis *et al.*, 1972).

3.6 Solvent Stripping form Miscella

Great care should be exercised to minimize the heat damage to the product during the desolventization of the miscella. Connel (1970b) has observed that excessive heating during removal of solvent from ginger extract can cause the formation of shogaol, zingerone, and aliphatic aldehydes. This can result in a decrease in pungency as well as the development of off-flavors. Thus, it is very necessary to control the temperature of the material carefully during distillation by use of vacuum. Constant stirring prevents overheating near the walls of the still. Volatile oil losses can be minimized by controlling the rate of distillation and by using tall columns. High essential oil content in oleoresin is necessary to get a good-quality oleoresin of the desired pleasant aroma. Chili oleoresin is, of course, and exception, since it does not contain any essential oil (Mathew *et al.*, 1971a; Lewis *et al.*, 1974).

Unlike pepper, there is no necessity for "contact time" in the case of chilies for maximum extraction of solids. The diffusion of solutes into the solvent is fairly quick. Handling of both chilies powder and extract requires special precautions, since they are highly pungent and irritate the skin and mucous membrane (Mathew *et al.*, 1971a).

The last traces of solvent are removed from oleoresin by vacuum distillation, followed by an azeotropic distillation technique.

4. Product Evaluation

The quality evaluation of oleoresins manufactured from major spices such as pepper, chilies, ginger, and turmeric have been studied, reviewed, and discussed by Todd (1960), Pruthi *et al.* (1960a), Ikeda *et al.* (1962), Stahl (1965, 1972), Pruthi (1968a), Anonymous (1970b), Nambudiri *et al.* (1970), Shankaranarayana *et al.* (1970), Mathew *et al.* (1971b). Lewis *et al.* (1972), Sabel and Warren (1972), Wijesekara *et al.* (1972), Govindarajan (1973), Govindarajan and Raghuveer (1973), Govindarajan *et al.* (1973), Ananthakrishna and Govindarajan (1974), and Govindarajan and Ananthakrishna (1974).

More than thirty-seven published reports (up to 1970) on the use of different techniques for the estimation of capsaicin in capsicums have been reviewed. Subsequently, Mathew *et al.* (1971b) achieved the separation of capsaicin from other constituents in the capsicum oleoresin by TLC. The method is claimed to be simple and convenient for adoption as a routine method, but Govindarajan and Ananthakrishna (1974) have since developed a simple, rapid, and accurate paper chromatographic method for the determination of capsaicin.

Govindarajan *et al.* (1973) also developed an interesting profile of ground black pepper through description fo the odor of the components from column fractionation of the essential oil and total oleoresin of pepper as put in final form by a trained panel at round-table discussions. It has been successfully applied to different varieties of Indian black pepper (Fig. 1) as well as to trade types and is claimed to be useful in the selection of new, superior varieties of pepper, in conjunction with objective data on color, size, pungent principles, and yield taken together.

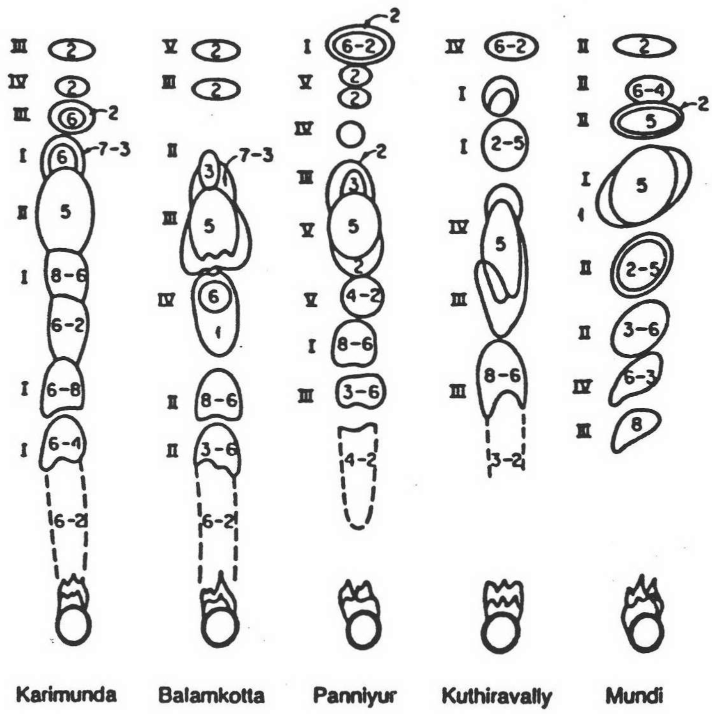

Figure 1 Tracing of TLC analysis of oleoresins from five varieties of Indian black pepper. From Govindarajan et al. (1973).

5. Forms or Types of Oleoresins

In some food applications, essential oils and/or oleoresins may be used "as is." However, because of their limited solubility, and the minute quantities required to produce strengths equivalent to those of natural spices, spice manufacturers usually further process the extractives into forms that are more convenient to use from the standpoint of both solubility and strength.

5.1 Liquid Forms of Oleoresins

Liquid forms of oleoresins and essential oils are available in both oil-soluble and water-dispersible types as follows :

a. Oil-Soluble Type.

In the oil-soluble type, the spice extractives are extended in a medium that will allow for quick and complete solubility in such products as shortenings, cake or cookie dough, icings, and any other food with high fat or oil content.

b. Water-Dispersible Form.

Oleoresins and essential oils are made water-dispersible by blending the extractives with a solubilizing agent such as Polysorbate 80. Solubilized spice preparations of this type may be added directly to water; they will spread readily throughout the vehicle, facilitating uniform dispersion of flavor and aroma in the finished food. Pickle packers, sausage makers, and soup and catsup processors often use this type of spice in their products.

5.2 Dry Form of Oleoresins

Dry form of oleoresins and essential oils consist of two types of products : (1) dry, soluble and (2) spray dried. Both forms are soluble or dispersible in the mediums in which they are intended to be used. They are discussed later in this chapter.

6. Oleoresin Pepper

Narayanan *et al.* (1964) reported a comparative study on different methods of extraction of oleoresin from pepper rejections as well as its storage stability. Natarajan *et al.* (1967b), while briefly reporting the preparation of oleoresin from black pepper, concluded that light pepper is the best raw material for its manufacture. Ziegler (1969) has patented the process for liquid pepper in Canada, West Germany, and the United States. Nambudiri *et al.* (1970) have described the factors affecting the manufacture of oleoresin of pepper. Shankaranarayana *et al.* (1970), Wijesekara and Jayawardena (1972), and Wijesekara *et al.* (1972) discussed the methods for the quality control of oleoresin of pepper. Govindarajan *et al.* (1973) developed a descriptive profile for pepper oleoresin, etc.

6.1 Botanical aspect of *Piper nigrum* Linn.

Piper nigrum Linn. (Fig. 2) is in the family of Piperaceae. It local names in various countries are pepper (English); pepe (Italian); pimenta (Spanish); poiure (French); prik-thai (Thai). The name "Pepper" is from the Sansakrit "Pippali". It means to fruit of *Piper nigrum* which has characteristic pungency and aroma (Standord, 1934).

Piper nigrum is native of southern India but is now cultivated extensively in the tropical areas including Thailand. The plant is a perennial, climbing shrub or vine with a smooth and woody. The plant clings to a tree or other support by means of numerous short rootlets produced at the joints of the stem. In its natural stalk, the plant may reach a height of 20 to 25 feet, but under cultivation it is usually kept down to 15 feet. The leaves are alternate, simple, ovate, dark green, entire with nearly rounded base and a some what acute tip. The flowers are very small, whitish, sessible, perfect and borne in pendulous, dense in spikes. The fruit is small, containing a single seed, sessile, nearly globular; at first green then yellowish and finally red when ripe. The fruit is borne on spikes 4 to 5 inch long, each spike carrying from 50 to 60 berries.

Piper nigrum requires a warm, humid, tropical climate, with heavy rainfall and intermittant spells of dry weather and some shade. The most suitalbe soil a well-drained vegetable loam. The land should be flat; hillsides, if used for the growing of pepper, should be terraced (Guenther, 1952).

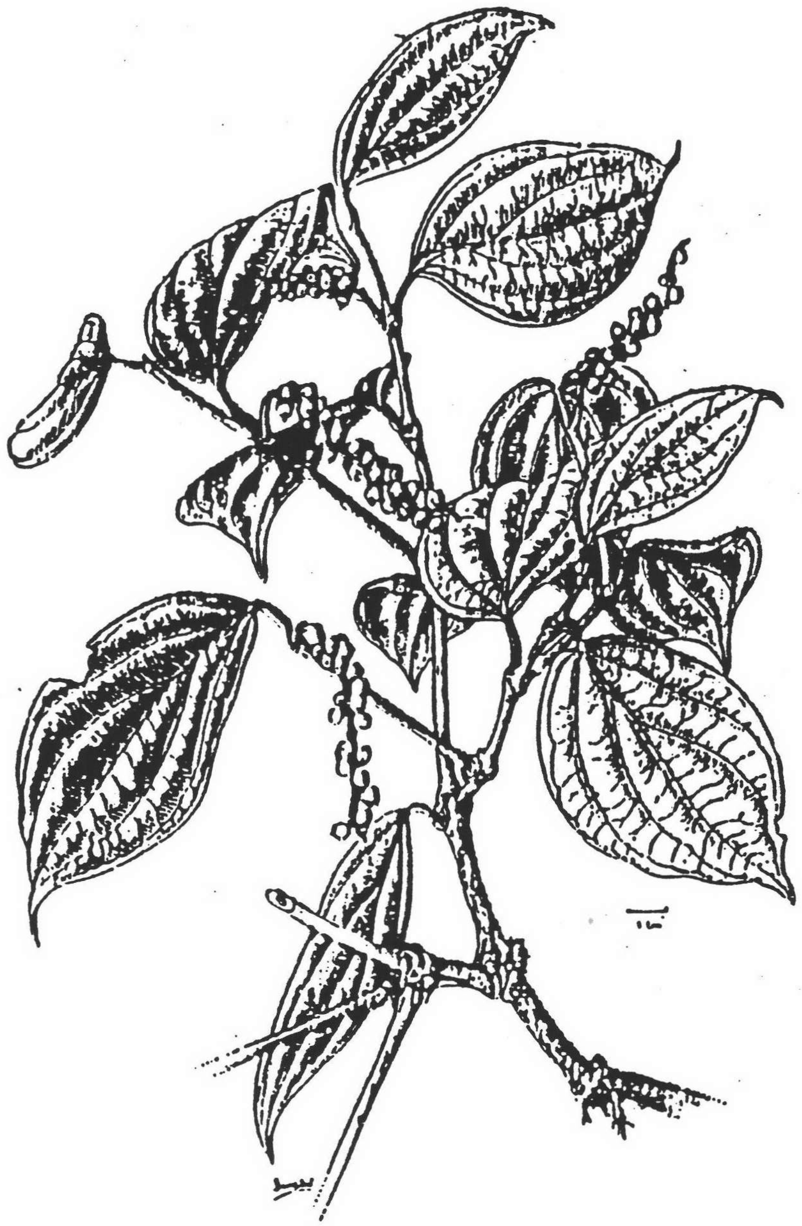

Figure 2 *Piper nigrum* L. (Piperaceae)

6.2 Principle uses

Pepper oleoresin is used chiefly in meat products, but also in virtually all kinds of savoury foods including pickles, sauces, gravies, dressings, (pet foods), chutneys, soups and many kinds of snacks such as savoury biscuits and crisps. Pepper oleoresin is produced and used in far greater quantities than any of the other oleoresins under consideration.

6.3 Consumption

It is estimated that between 60 and 65 tons of pepper oleoresin are currently used each year in the United Kingdom.

6.4 Prices

Imported pepper oleoresin (from the United States) : current price $15.00/kg c.i.f. (ex-duty at 10%), or about £ 6.60 duty paid.

United Kingdom produced pepper oleoresin, dispersed :

Base	July 1972	May 1973
(£ /kg, prices for orders of 50 kg, delivered)		
Salt	0.50	0.56
Flour	0.54	0.60
Rusk	0.55	0.61
Dextrose	0.56	0.62

6.5 Yield

It is reported that the yield of oleoresin from black pepper varies between about 10 and 15 per cent. One company indicated that it considered an economic yield to be 12.5 per cent.

6.6 Standards, Specifications and Description

No United Kingdom standards exist for black pepper oleoresin.

Black pepper oleoresin imported into the United Kingdom from the major United States supplier is described as follows:

Oleoresin black pepper. Raw material : Ground berries, *Piper nigrum* L., conforming to all provisions of the Federal Food, Drug and Cosmetic Act.

Oleoresin qualifications

- **Volatile oil :** (Modified Clevenger Method, ASTA Analytical Method #5.0) 18-20%
- **Physical characteristics :** A thick liquid, usually pourable at room temperature when thoroughly mixed, requiring mixing to ensure uniformity of withdrawal. When dispersed on salt or sugar at 2% by weight, the colour shall be an olive-brown to green.

  Piperine by Kjeldahl : 50-55%

  Piperine by UV Spectrophotometry : 35-40%
- **Aroma and flavour :** The aroma shall be characteristic of black pepper, slightly aromatic, with an underlying musty odour. The flavour shall be slightly warm and pleasant initially, followed by a very pungent, biting sensation. The flavour shall conform to standards established for test panel.
- **Replacement strength :** 1 1b of oleoresin black pepper will replace 20-25 lb of a good grade of ground black pepper.
- **Food additives :** The high viscosity of black pepper oleoresin obtained from extraction necessitates the addition of approved food additives mono- di- and triglycerides to ensure ease of application in seasoning.

- **Residual solvent :** Less than 30 ppm total as determined by the standard procedure described in "The Estimation of Residual Solvent in Spice Oleoresins," Food Technology, 1960, XIV, 301.

There is also a United States standard specification for oleoresin black pepper, published by the Essential Oil Association of the USA (EOA No. 240). Oleoresin imported into the United Kingdom from United States producers conforms to this specification.

7. Chemical constituents of Oleoresin

Oleoresin contains the volatile aroma constituents and non-volatile pungent principles. It will also contain all the colouring principle present in the spice. Therefore oleoresin represents all that is good in the spices. Besides the significant constituents, oleoresin will also contain other constituent like pigments, fixed oil, sugars, resins etc. The proportion of these will depend upon solvent used for extract.

Peper oleoresin is the concentrated essence of black pepper. Piperine provides the intense pungency while pepper oil constituents contribute to the aroma. The dark green colour is due to chlorophyll of the outer skin and the black enzymatically oxidised phenolic compounds present in the skin.

8. Piperine

Structure and chemical properties

Piperine (1-[5-(1,3-Benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine; 1-piperoyl piperidine; $C_{17}H_{19}NO_3$: MW 285.33) (Fig. 3)

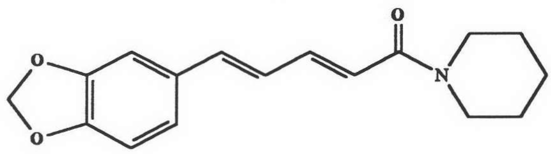

Figure 3 The structure of piperine

The piperidine alkaloid piperine was first isolated from *Piper nigrum* in 1820 (Purseglove, 1981). It was later isolated from other *Piper* spp. (Table 2). It has also been found in *Psilocaulon obsimile* (Family Aizoaceae), in fruits of *Xylopia brasiliensis* (Family Annonaceae) (Darnley, 1974) and in leaves of *Rhododendron fauriae* var. *rufescens* (Family Ericaceae) (Kawaguchi, 1942). On hydrolysis by acid or alkaline, piperine decomposes into strongly basic piperidine and piperic acid (Fig. 5) (Cluenther, 1952). Piperine can be photoisomerized into a mixture other configuration (Grewe *et al.*, 1970).

Table 2 Piperine in *Piper* spp.

Piper	Plant part	Reference
Piper album Vahl.	fruit	Glasl *et al.*, 1976
P. chaba Hunter	stem	Bose, 1935
P. cubeba Linn.	fruit	Hodorn and Jungkun, 1951
P. quineense Schum & Thonn	root	Addae-Mensah et al., 1977; Dwuma-Badu *et al.*, 1976; Okogum and Ekong, 1974
	fruit	Addae-Mensah *et al.*, 1977.

Table 2 (Continued)

Piper	Plant part	Reference
P. longum Linn.	root	Dutta *et al.*, 1977.
	fruit	Dhar and Atal, 1967;
		Govindachari *et al.*, 1969
P. nepalense Miq.	stem	Gupta *et al.*, 1972
P. nigrum Linn.	fruit	Nakatani and Inatani, 1981;
		Verzele and Qureshi, 1980
	stem	Singh *et al.*, 1976
	seed	Traxler, 1971
P. novae-hollandiac Miq.	stem	Loder *et al.*, 1969
P. peepuloides Roxb.	fruit	Dhar and Raina, 1973
P. retrofractum Vahl.	stem	Mishra and Tewari, 1964
P. sylvaticum Roxb.	root	Barnerji and Dhara, 1974

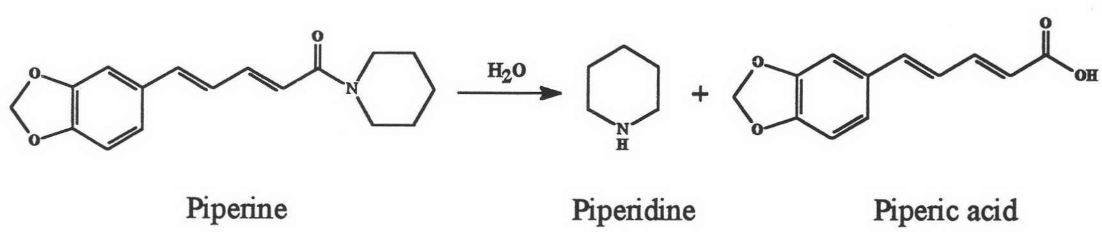

Figure 4 The hydrolysis of piperine.

For the physical properties, piperine has crystalline in monoclinic prism melting at 129-130°C, tasteless at first but burning after taste. It is neutral to litmus (pK (18°C) = 12.22; K=6x10^{-10}, almost insoluble in water and petroleum ether but readily soluble in alcohol, chloroform, ether, benzene and acetic acid (Budavari, 1989).

9. Pepper oil

- Composition of pepper oil

The volatile pepper oil is obtained by steam distillation. It is an almost colorless to bluish-green liquid with a characteristic odour recalling that of whole pepper. The physical and chemical constants of pepper oil are described below (Budavari, 1989).

Specific gravity at 15°C	0.890-0.900
Refractive index at 20°C	1.4935-1.4977
Optical rotation at 25°C	-3° to -5°
Solubility 1:5 in 90% alcohol	

A study on the composition in pepper oil was begun during the last century. The pepper oil is primary a complex mixture of hydrocarbon such as monoterpenes (50-80%), sesquiterpenes (20-40%) and small amounts of oxygenated terpene compounds (less than 4%) (Hasselstrom *et al.*, 1957; Ikeda *et al.*, 1962: Jennings and Wrolstad, 1961; Nigam and Handa, 1964; Richard *et al.*, 1971; Wrolstad and Jennings, 1965). The presence of terpenoids in black pepper oil including terpene hydrate, α-phellandrene and β-caryophyllene was reported over 90 years ago (Schreiner and Kremers, 1901). Later, a number of additional substances were identified including those shown in Table 3.

Pepper oils from different cultivars have their own characteristics odour due to the different proportion of their volatile components. The aromatic characters of the component present in black pepper oil are shown in Table 4 (Lewis *et al.*, 1969b).

Table 3 Constituents identified in black pepper oil

Constituents	Reference
Monoterpene hydrocarbons	
Camphene	Ikeda *et al.*, 1962
Δ^3-Carene	Wrolstad and Jennings, 1965
p-Cymene	Wrolstad and Jennings, 1965
Limonene	Hasselstrom *et al.*, 1957
Myrcene	Wrolstad and Jennings, 1965
cis-Ocimene	Ikeda *et al.*, 1962
α-Phellandrene	Hasselstrom *et al.*, 1957
β -Phellandrene	Ikeda *et al.*, 1962
α-Pinene	Hasselstrom *et al.*, 1957
β-Pinene	Hasselstrom *et al.*, 1957
Sabinene	Ikeda *et al.*, 1962
α-Terpinene	Ikeda *et al.*, 1962
γ-Terpinene	Ikeda *et al.*, 1962
Terpinolene	Wrolstad and Jennings, 1965
α-Thujene	Wrolstad and Jennings, 1965
Sesquiterpene hydrocarbons	
α-cis-Bergamotene	Russell *et al.*, 1986
α-trans-Bergamorene	Russell *et al.*, 1986
β-Bisabolene	Russell *et al.*, 1986
δ-Cadinene	Muller *et al.*, 1968
γ-Cadinene	Debrauwere and Verzele, 1976
Calamenene	Debrauwere and Verzele, 1976
β-Caryophyllene	Nigam and Handa, 1964
α-Copaene	Richard *et al.*, 1971
α-Cubebene	Richard *et al.*, 1971

Table 3 (continued)

Constituents	Reference
β-Cubebene	Artem'ev and Mistryukov, 1979
ar-Curcumene	Russell and Jennings, 1969
δ-Elernene	Debrauwere and Verzele, 1976
β-Elemene	Russell and Else, 1973
β-Farnesene	Russell and Else, 1973
α-Guaiene	Debrauwere and Verzele, 1976
α-Humulene	Muller *et al.*, 1968
Isocaryophyllene	Muller *et al.*, 1968
γ-Murrolene	Muller *et al.*, 1968
α-Santalene	Muller *et al.*, 1968
α-Selinene	Lewis *et al.* 1969a
β-Selinene	Lewis *et al.* 1969a
α-Patchoulene isomer	Artem'ev and Mistryukou, 1979
Oxygenated monoterpenes	
Borneol	Debrauwere and Verzele, 1975
Camphor	Debrauwere and Verzele, 1975
Cavacrol	Debrauwere and Verzele, 1975
cis-Carveol	Russell and Jennings, 1969
trans-Carveol	Russell and Jennings, 1969
Carvone	Russell and Jennings, 1969
Carvetonacetone	Debrauwere and Verzele, 1975
1,8-Cineole	Debrauwere and Verzele, 1975
Cryptone	Russell and Jennings, 1969
p-Cymene-8-ol	Russell and Jennings, 1969
p-Cymene-8-methyl ether	Debrauwere and Verzele, 1975
Dihydrocarveol	Hasselstrom *et al.*, 1957
Dihydrocarvone	Debrauwere and Verzele, 1975

Table 3 (continued)

Constituents	Reference
Linalool	Russell and Jennings, 1969
Myrtenol	Debrauwere and Verzele, 1975
cis-Sabinene hydrate	Russell and Jennings, 1970
trans-Sabinene hydrate	Russell and Jennings, 1970
β-Pinone	Debrauwere and Verzele, 1975
1-Terpinen-4-ol	Richard and Jennings, 1971
Oxygenated monoterpenes	
1-Terpinen-5-ol	Debrauwere and Verzele, 1975
α-Terpineol	Richard and Jennings, 1971
Phenyl ethers	
Eugenol	Richard and Jennings, 1971
Methyl eugenol	Richard and Jennings, 1971
Myristicin	Richard and Jennings, 1971
Safrole	Richard and Jennings, 1971
Anethole	Artem'ev and Mistryukov, 1979
Oxygenated sesquiterpenes	
β-Caryophyllene alcohol	Debrauwere and Verzele, 1975
Caryophyllene ketone	Richard *et al.*, 1971
Caryophyllene oxide	Debrauwere and Verzele, 1975
Epoxy-dihydrocaryophyllene	Hasselstrom *et al.*, 1957
Nerolidol	Richard and Jennings, 1971
Eudesmol	Artem'ev and Mistryukov, 1979
Miscellaneous	
Butyric acid	Debrauwere and Verzele, 1975
Hexanoic acid	Debrauwere and Verzele, 1975
Benzoic acid	Debrauwere and Verzele, 1975
Cinnamic acid	Debrauwere and Verzele, 1975

Table 3 (continued)

Constituents	Reference
Piperonic acid	Debrauwere and Verzele, 1975
Piperonal	Debrauwere and Verzele, 1975
Piperidine	Hasselstrom *et al.*, 1957

Table 4 Aromatic attributes of the components of black pepper oil

Component	Main Character	Subsidiary Attributes
Monoterpene hydrocarbons		
α-Pinene	Pine-like	Warm, Resinous, Refreshing
Camphene	Camphoraceous	Oily, Terpeney
Sabinene	Peppery Warm,	Woody, herbaceous
β-Pinene	Dry woody	Resinous, Pine-like, Terpeney
Myrcene	Sweet balsamic	Resinous, Lemony, Fresh
α-Phellandrene	Peppery	Woody, Fresh, Citrus, Minty
Δ^3-Carene	Penetrating	Sweet, Irritating
α-Terpinene	Lemony	Fresh
β-Phellandrene	Peppery Minty,	Slightly citrus-like
Limonene	Fresh Light,	Orange-like
γ-Terpinene	Herbaceous	Warm, Lemony
Terpinolene	Sweet piny	Slightly anisic
Sesquiterpene hydrocarbons		
β-Caryophyllene	Woody spicy	Dry, Clove-like
β-Farnescene	Sweet	Warm
Humulene	Sweet woody citrus	Penetrating
β-Selinene	Sweet woody	Peppery
α-Selinene	Herbaceous	Warm, Woody, Peppery
β-Bisabolene	Warm spicy	Balsamic, aromatic
Oxygenated compounds		
Linalool	Floral woody	Light, Refreshing, Slightly citrus-like
l-Terpinen-4-ol	Warm pepery	Earthy, Woody
Carvone	Warm herbaceous	Spicy, Slight floral
Caryophyllene ketone	Fruity	Minty