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The Director
ICAR-Indian Institute of Spices Research
Marikunnu P.O, Kozhikode 673012

भाकृअनुप ICAR

Ph: 0495-2731410 | E-mail: directorspices@gmail.com

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ICAR - Indian Institute of Spices Research
Kozhikode-673012, Kerala

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Authors
Jayashree E
Alfiya P V
Anees K
Archana R

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Jayashree E
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Essential Oil Extraction From Spices

Spices are known for their bioactive compounds and have drawn attention in the recent past for their nutraceutical properties. Spice of commerce are obtained from different plant parts such as root, stem, leaves, fruits, flowers *etc.* and in various forms like the whole spices, roasted, paste, crushed, powdered and extractives. Compared to the various forms of spices, spice extractives have better stability in terms of flavour and storage. The important two categories of extractives include, essential oils and oleoresins. In order to recover these extractives, cell matrix of the respective spice has to be disrupted. Generally, dried spices are used for the extraction purposes compared to fresh spices as they have better stability and are more concentrated. Dried material is crushed so that surface area is increased and solvent can very well diffuse into the cell matrices. Efficiency of extraction depends upon the extraction time, solvent used, temperature of extraction and solvent-solid ratio.

Essential oils are volatile aromatic liquid oils extracted traditionally by hydro or steam distillation of spices. They are complex mixtures of low molecular weight compounds and are responsible for the aroma produced from any spice. There are approximately about 3000 essential oils being extracted and among this around 300 are of commercial importance. Essential oil does not represent a spice completely as they contain only volatile components. Distillation is considered as the conventional method for extraction of essential oils.

Oleoresins are the non-volatile part of any spice and they are highly concentrated with wholesome flavour and aroma of the respective spice. Compared to fresh and ground spice, oleoresins are considered more hygienic. Extraction of oleoresin involves the use of various solvents and acetone is the most commonly used one. Solvent remaining in the extract after the extraction is removed by evaporation or distillation. Solvent extraction is the conventionally followed method for the extraction of oleoresin.

Sources of natural essential oil

Essential oils are generally derived from one or more plant parts, such as flowers (e.g. rose, jasmine, carnation, clove, mimosa, rosemary, lavander), leaves (e.g. mint, *Ocimum* spp., lemongrass, jamrosa), leaves and stems (e.g. geranium, patchouli, petitgrain, verbena, cinnamon), bark (e.g. cinnamon, cassia, canella), wood (e.g. cedar, sandal, pine), roots (e.g. angelica, sassafras, vetiver, saussurea, valerian), seeds (e.g fennel, coriander, caraway, dill, nutmeg), fruits (bergamot, orange, lemon, juniper), rhizomes (e.g. ginger, calamus, curcuma, orris) and gums or oleoresin exudates (e.g. balsam of Peru, myrrh, benzoin/benjamin).

Method of Extracting Essential Oil

The essential oil industry uses different terminologies to differentiate the method of Hydro-distillation, the most commonly followed method for extraction of essential oil from aromatic plant materials: water distillation; water and steam distillation; and direct steam distillation. Theoretical considerations of all these three methods are same wherein, distillation of two-phase systems is dealt with. Only the manner of handling the material varies among methods. Even though, hydro-distillation is the most common methods of essential oil extraction, there are some special case variants to this method depending on the nature of the plant matrix used for extraction. In case of Lemon oil and orange oil, extracted from their fruit peel, a process of expression is applied. It involves puncturing the oil glands by rolling the fruit over a trough lined with sharp projections that are long enough to penetrate the epidermis. In case of essential oils extraction from flower petal, where the oil content is too small, enfleurage is the process of extracting essential oil. This involves layers of fat packed with flower petals for hours for absorption of aromatic oil to the fat, which then extracted with alcohol. This old method of perfumery industry has now been replaced with extraction with organic solvents such as petroleum ether and hexane. This has an advantage of maintaining a uniform temperature (about 50°C) which gives extracted oil a natural odour.

Hydro-Distillation

Addition of sufficient quantity of water to a still packed with the plant material and heating the same to boil is the most common practice for hydro-distillation. Live steam injection into the plant charged still is another mode of operation. The hot water and steam forces the essential oil to move out of the plant matrix (oil glands). The vapour mixture thus release contain water and oil, is then condensed by indirect cooling with water. A separator collects the distillate, where oil separates based on its specific gravity from the distillate water.

Mechanism of Distillation

Hydro distillation of any plant material involves the following main physicochemical processes:

i) Hydro-diffusion

ii) Hydrolysis

iii) Decomposition by heat

i. **Hydro-diffusion**

Diffusion of essential oils and hot water through plant membranes is known as hydro-diffusion. The dry plant material is size reduced to enable the dry steam to penetrate easily into the oil bearing cells of the plant material and to free the volatile oil. Under normal conditions, the membranes of plant cells are almost impermeable to volatile oils for its release. But at boiling temperature of water, a part of the volatile oil present within the oil gland dissolves in the water, swells up the membrane due to osmotic exchange of this oil-water solution, and escapes to outer surface, where the oil is vaporized by passing steam. The speed of oil vaporization is by their degree of solubility in water, not by the volatility of the oil components. Therefore, low boiling but less water-soluble constituents are distilled after the high-boiling but more water-soluble constituents of oil in plant tissue. This is the reason why distillation of whole material takes longer time than size reduced material.

ii. Hydrolysis

Hydrolysis refers to the chemical reaction between water and the components of essential oils. At high temperatures, the ester form of essential oils constituents tend to react with water to produce alcohol and acids but the reaction is not complete in all directions due to the limitation of water access.

In water distillation method, increasing the amount of water results in increased amount of alcohol and acids. Leading to low essential oil yield. Hydrolysis primarily have a direct correlation with the time of contact between water and oil. This is considered as one of the disadvantages of the water distillation.

iii. Decomposition by heat treatment

Almost all constituents of essential oils are unstable at high temperature. Hence low temperature distillation is preferred for obtain the best quality oil. In steam distillation, the temperature is determined by the operating pressure while, water distillation and water & steam distillation operates at atmospheric pressure. These three effects, *viz.* hydro-diffusion, hydrolysis and thermal decomposition do occur simultaneously and affect one another. The rate of diffusion and the solubility of oil in water increases with temperature. The same holds true for the extent and rate of hydrolysis. The three basic principles to obtain better oil yield are: (1) maintain the operating temperature as low as possible, (2) in the case of steam distillation, use as little water as possible, and (3) comminute (pulverise) the plant material and pack it uniformly in the still before distillation.

Types of Hydro-Distillation

Three are three types of hydro-distillation for isolating essential oils from plant materials:

1. Water distillation
2. Water and steam distillation
3. Direct steam distillation

1. Water distillation

Completely immersed plant material in water is boiled by direct fire/steam jacket/closed steam jacket/closed steam coil/open steam coil. The main feature of this method is that there is direct contact between boiling water and plant material. Overheating has to be avoided during the process which can be taken care if jacket or closed steam coil is used for boiling water. Proper agitation during boiling is must to avoid agglomerations of dense material on bottom of the still which might result in thermal degradation. This can be achieved by loosely packing the plant material in the still. In case of cinnamon bark, which are rich in mucilage, it is advised to use after powdering to help it for better dispersion in wtare. The leached mucilage might lead to increased viscosity and charring. Therefore, it is better to carry out a lab-scale water distillation using Clevenger system to assess the changes during the distillation process and to estimate oil yield of the test material. Advantages of water distillation are: avoids the chances for forming lumps; stills used are inexpensive; easy to construct and suitable for field operation.

Disadvantages of water distillation

- Estric componets of oil tends to hydrolyse forming alcohole and acids during water distillaiton. acyclic monoterpene hydrocarbons and aldehyde components of oil tend to polymerize under decreased pH of water during distillation
- The solubility of phenolic fraction of oil in water makes the complete removal difficult
- Since the water distillation engages one or two persons, it takes a long time to harvest more much oil. Hence chances of mixing good quality oil with bad quality oil
- water distillation is comparatively a slower process than either water and steam distillation or direct steam distillation

2. Water and steam distillation

In water and steam distillation, the steam used for distillation is generated either by a satellite boiler or within the still as a separate chamber. The machinary used is somewhat similar to water distillation system except that a perforated grid supports the plant material above the boiling water. All rural people engaged in water distillation eventually shifts into water and team distillation system due to the better quality of the obtained oil, with minor modification to their existing system. In case of insufficient quantity of water for complete distillation, a re-circulating (cohobation) tube is used for adding condensate water back to the still.

Advantages of water and steam distillation

- Higher oil yield compared to water distillation
- Components of the volatile oil are less susceptible to hydrolysis and polymerization
- If refluxing (cohobation) is managed properly, then the loss of polar components of essential oil can be minimized
- It provides more reproducible quality of extracted oil
- Steam and water distillation is faster than water distillation, so it is more energy efficient

Disadvantages of water and steam distillation

- Oils of high-boiling range necessitates larger quality of steam for vaporization due to the low pressure of rising steam
- As the distillation progresses, the plant material in still becomes wet, which eventually slows down distillation
- A baffle is required to avoid waterlogging and direct contact with vigorously boiling water with the lower plant material resting on the grid

3. Direct steam distillation

Steam distillation uses steam generated outside the still in a boiler for distilling plant material. The steam is fed through an inlet to the still packed with plant material supported on a perforated grid. Since the atem is generated outside the still, the amount of steam can be easily controlled and one can make sure that the plant material is not heated to temperature higher than 100°C, resulting in least degree of thermal degradation. The steam, as it rises to the top, carries the essential oil and the mixture of steam and oil is then cooled in a condenser. The condensate is collected in a separator where the oil is decanted based on its specific gravity. Steam distillation is the most widely accepted process for the production of essential oils on large scale.

Essential oil extraction unit

Specifications of the essential oil extraction unit (Pilot plant) at ICAR-IISR

S. No.	Details of the Unit		Specifications
1.	**Charging vessel or still**		
	Capacity	:	150 litres
	Type	:	Cylindrical, vertical, top conical, bottom dished
	Material	:	SS-304, 3 mm thick sheet for the shell
	Fitting and accessories	:	• Fully open type with flange joint, Charging hole, discharge hole, sight glass, grill, perforated sheet, nozzles etc.

			• Mounted on lugs SS 202.
			• Top temperature gauge, pressure release valve, safety valve and steam inlet nozzles with valves
2.	**Coil Type Condenser**		
	Capacity	:	2 m^2 Heat Transfer Area
	Type	:	Box Type, Vertical
	Material	:	• SS-304, 3 mm thick sheet, 1" × 1.6 mm thick seamless pipes for the tubes and SS header
			• SS-304, 6 mm sheet for the tube plate
3.	**Oil Water Separator**		
	Capacity	:	25 Litres
	Type	:	Box Type, Vertical
	Material	:	SS-304, 1.6 mm thick sheet for the shell
4.	**Electric Boiler**		
	Capacity	:	27 kg/h of steam
5.	**Structure and Pipe lines**		
	Structure	:	SS-202
	Internal pipe lines	:	SS-304

Essential oil extraction from cinnamon leaves

Cinnamon leaves, derived from *Cinnamomum verum*, are a valuable source of essential oil, primarily rich in eugenol, which imparts its characteristic aroma and therapeutic properties. The extraction of essential oil from cinnamon leaves is typically performed using hydro-distillation or steam distillation, with the latter being preferred for its efficiency and quality. The process involved disrupting the oil-bearing cells in the leaves to release volatile compounds, which were then collected through condensation and separation. The pilot plant at ICAR-Indian Institute of Spices Research (IISR), Kozhikode, is designed to optimize this process, ensuring high yield and quality of cinnamon leaf oil for commercial and research applications.

Process of cinnamon leaf oil extraction

Preparation of Plant Material: Fresh or dried cinnamon leaves were collected, cleaned, and scrushed to increase surface area, facilitating the release of essential oils from oil glands. **Charging the Still:** The prepared leaves were loaded into the charging vessel, a 150-L stainless steel (SS-304) cylindrical still with a conical top and dished bottom, equipped with a perforated grill to support the plant material.

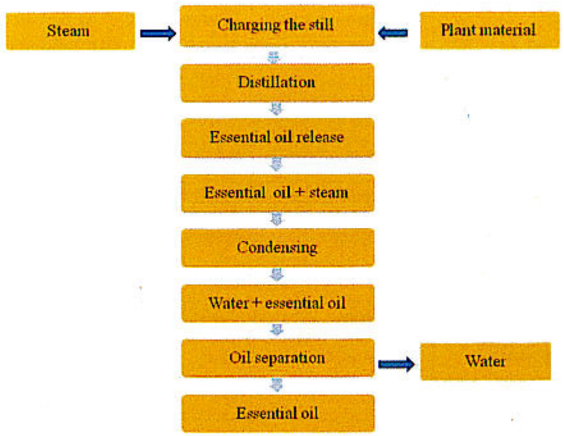
Steam Distillation: Steam was generated by a 27 kg/h electric boiler, is introduced into the still through inlet nozzles. The steam penetrated the leaf matrix, evaporating the essential oil. The steam-oil vapor mixture rose up and was directed to the condenser.

Condensation: The vapor mixture passed through a coil-type condenser (SS-304; 2 m^2 heat transfer area) where it was cooled by indirect water circulation, condensing into a liquid distillate containing water and essential oil.

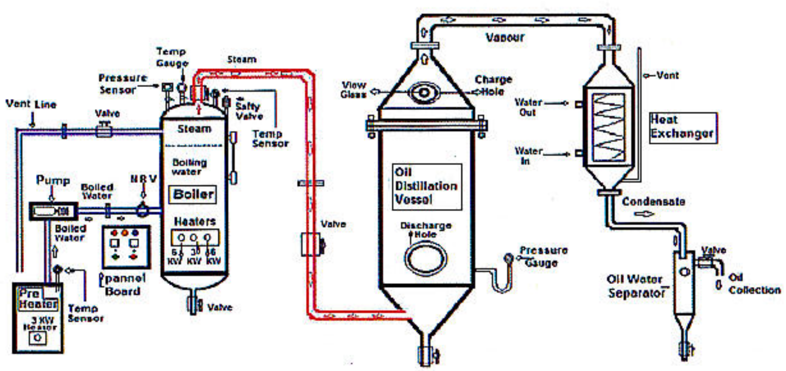
Separation: The distillate was collected in a 25 L oil-water separator (SS-304), where the cinnamon leaf oil was separated based on its specific gravity. The oil was then decanted for further processing or storage.

Essential oil content in some of the commonly used spices

Scientific name	Common name	Essential oil, %	Major active compound of essential oil
Allium sativum	Garlic	0.1-0.25	Allicin
Capsicum annum	Chilli	1-3	3-methyl butanal
Cinnamomum tamala	Bay leaf / Tejpatt	0.8-3.0	Eugenol
Cinnamomum verum	Cinnamon	0.4-3.0	Cinnamaldehyde
Coriandrum sativum	Coriander	0.03-3.0	Linalool
Cuminum cyminum	Cumin	2-5	Cuminaldehyde
Curcuma longa	Turmeric	15-25	α-turmerone
Elettaria cardamomum	Cardamom	11.0	1,8-cineole
Foeniculum vulgare	Fennel	2-3	Anethole
Mentha piperita	Pepper mint	0.5-4	Menthol
Murraya koenigii	Curry leaves	0.5	α-pinene
Myristica fragrans	Nutmeg	6-16	Myristicin
Piper nigrum	Black pepper	1-4	α-pinene
Syzygium aromaticum	Clove	15-20	Eugenol
Trigonella foenum-graecum	Fenugreek	5.55	Trigonellin
Zingiber officinale	Ginger	1-3	Zingeberene

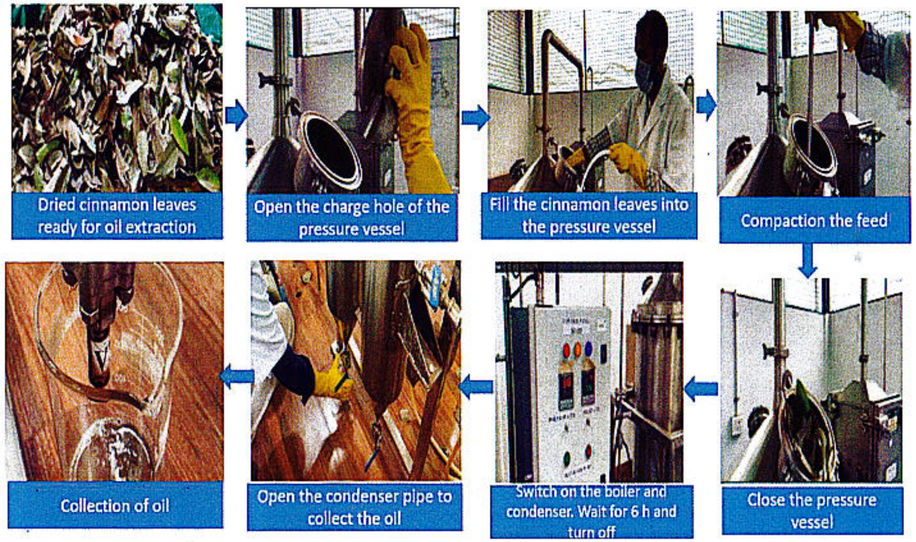


Flow chart for production of essential oil



Schematic diagram of essential oil extraction unit at ICAR – IISR, Kozhikode

Pilot plant for essential oil extraction



Process flow chart for cinnamon leaf oil extraction

The extraction of essential oils from spices represents a vital aspect of spice processing, harnessing the potent bioactive compounds that contribute to their flavor, aroma, and nutraceutical properties. Techniques such as hydro-distillation, water and steam distillation, and direct steam distillation offered diverse approaches to isolate these valuable extractives. The selection of extraction method is influenced by factors such as plant material, solvent type, extraction time, and temperature, significantly impacts the yield and quality of the essential oils. The essential oil extraction unit at ICAR-Indian Institute of Spices Research, Kozhikode, provides advanced infrastructure for large-scale production of essential oil, ensuring high-quality products with minimal thermal degradation. By optimizing extraction processes of spices, the industry can continue to meet growing commercial and therapeutic demands thereby preserving the essence of spices for culinary, medicinal, and industrial applications.

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