June 2010 ISSN : 0974 - 7443 Volume 5 Issue 1
CHEMICAL TECHNOLOGY
An Indian Journal
Trade Science Inc.
Review
CTAIJ 5(1) 2010 [19-24]

A brief review on essential oil extraction and equipment

S.H.Jain*, G.Ravikumar
Chemistry of Forest Products Division, Institute of Wood Science and Technology, Malleswaram, Bangalore - 560 003, (INDIA)
E-mail: shjain@icfre.org
Received: 2nd January, 2010 ; Accepted: 12th January, 2010

INTRODUCTION

India has rich bio diversity and geo climatic conditions for essential oil bearing crops. Out of 18000 plants species found in India, about 1500 are known to be aromatic and out of this around 150 to 200 species are commonly traded on World markets. A majority of them are obtained from agricultural lands but some 26 essential oils are obtained from wild/cultivated plants of forest origin. Eg. Cedarwood, Citronella, Lemongrass, Patchouli, Davana, Eucalyptus, Sandalwood, Cinnamomum, Cassia etc. World total production of essential oils is estimated at about one lakh ton and India shares about 20% therein.

The ancient Arabian people began to study the chemical properties of essential oils & developed and refined the distillation process. The first pure distillation procedures for producing fully purified chemical substances were carried out by Arab Muslim chemists for industrial purposes such as isolating natural esters (perfumes) and producing pure alcohol. Distillation was later known to Greek alchemists from the 1st century AD, and the later development of large scale distillation apparatus occurred in response to demands for spirits[3].

Essential oils are concentrated, fragrant, odiferous steam volatile plant essences obtained by steam/water distillation of the plant material. They are secondary plant metabolites occur in different parts of the plant: ideally they are isolated with minimum chemical changes from human intervention. Eg. bark (Cinnamomum), flowers (Jasmine), leaves (Eucalyptus), berries (Bursera), grasses (Palmarosa), roots (vetiver), herbs (Patchouli) and wood (Sandal) etc. Each essential oil is composed of different chemical components. These various components combine in different ways to form specific oils of very distinct note. No two essential oils are alike in their composition or effects. Ideally a perfume should evaporate smoothly from the top note to the bottom note with the main theme apparent at every stage.

Techniques of distillation of aroma oils

Distillation of aromatic herbs implies vapourizing or liberating the oils from the trichomes/plant cell membranes of the herb in presence of high temperature and moisture and then cooling the vapour mixture to separate out the oil from water. It is the most widely used and cost effective method in use today for producing majority of the essential oils throughout the World.

BASIC PRINCIPLE INVOLVED IN THE PROCESS

Thermodynamics play an important role in distillation of essential oils. In order to vapourize any liquid, energy in the form of latent heat must be applied. The fundamental nature of steam distillation is that it enables a compound or mixture of compounds to be distilled at a temperature substantially below that of the boiling point of the individual constituent. In the presence of steam, the components of essential oil are volatilized at a temperature close to 100°C at atmospheric pressure. The mixture steam along with hot vapours are allowed to

Review

pass through a cooling system, condenses to form a liquid in which the oil and water comprise two distinct layers. Most of the essential oils are lighter than water and form the top layer. The steam that is used for distillation is generated either within the steel vessel that contains the plant material or by an external boiler.

Essential oils can be extracted using a variety of methods, although some are not commonly used today. Commercially, the most commonly applied method for extraction of essential oil is steam distillation. Several processes like hydro-distillation, water-steam distillation, steam distillation, maceration, hydro-diffusion, enfleurage, expression and solvent extraction are available for the extraction of aroma substances.

Application of these processes, either singly or in combination, depends upon the nature of the raw material and of the essential oil intended to be recovered. The following different methods are widely applied for extracting essential oil from different parts of plant depending upon the raw material to get optimum yield.

Normal methods of distillation

There are 3 different methods of essential oil distillation viz., Hydro (water) distillation, water-steam distillation, and steam distillation.

Hydro distillation

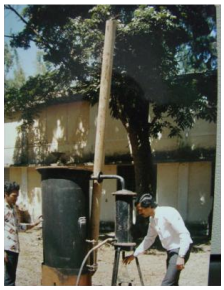
Since pre historic day's hydro-distillation is extensively used and it is the versatile method for extraction of essential oils. The main feature of this method is that the material is immersed and is in direct contact with the boiling water. The essential oil along with steam is condensed through a condensation system and the oil floats on top is separated. Hydro distillation process is normally used for quantification of essential oils at laboratory level using Clevenger apparatus. The system is heated only with the help of electric mantle. The long vertical glass tube and condenser are made up of glasses which require careful handling. The risk involved in the process is that the still can run dry, or be overheated, burning the aromatics and affecting quality of essential oil.

Water steam distillation

This method is employed where the perfumery material is vulnerable to direct steam. Here the plant material will not be in direct contact with water. Water is boiled below and the steam passes through a bed of plant material kept on a false bottom in a still or retort. The conventional distillation units mostly work on the principle of steam distillation are installed in places where water and fuel resources are easily available. But high cost of transportation of bulky raw material from field to industry and other constraints of local need have hindered installation of more distillation units. Keeping these things in mind and to meet the needs of farmers and small scale distillers, the Institute of Wood Science and Technology (IWST), Bangalore has developed a compact, portable water-steam distillation unit for oil distillation in the field. This unit has been designed and fabricated for distillation of oils from leaves and other aromatic plant material.

Portable distillation unit developed by IWST, Bangalor

The portable field distillation unit (250-300 L. capacity) is made of mild steel with detachable parts for easy transportation from place to place[7]. This can be set up easily in field even limited water resource for condensation and other utility purposes. The unit comprises of a still with a lid on top and vapour outlet at the side. The material to be distilled is placed over the perforated plate inside the still and is heated from a fire box (oven) fixed below, using locally available fuel wood. The fire box has an efficient outlet for smoke discharge. The condenser part of the unit is of vertical type made of mild steel with suitable inlet and outlet for water circulation. It has been provided with five parallel stainless steel tubes inside to increase the area of condensation. The oil-water vapour emerging out of the still enters all the five steel tubes instead of one and gets condensed faster and more efficiently to yield 30% more oil. The distillate can be collected in any suitable vessel and the oil which floats on top can be separated easily.



Advantages of portable distillation unit

- The economics of essential oil production depend on both the quality cultivation of the plant material and its efficient extraction of oil.
- Conventional stationary distillation units are mostly installed in a place where water and fuel resources are in abundance.
- High cost of transportation of the bulky raw material from field to distillation site and storage of raw material add to the increased cost of distillation.

This portable distillation still being portable in nature is easier to transport to the field and the simplicity of operation reduces the efforts and cost of operation. Economics of operation will depend on the availability and nature of raw material, operations conditions and market requirement.

Steam distillation

Steam distillation is a method for distilling compounds which are heat-sensitive. In this method, steam is generated separately in a boiler is passed directly to the plant material kept on a grid in a still. The vapour mixture is cooled and condensed and the oil is separated as usual. Industries processing tons of material employ steam distillation process using steam pressure around 20 to 40psi and temperature rising above 150°C. Consequently, the plant material is supported on a perforated grid or screen inserted at some distance above the bottom of the still. The lower part of the still contains water upto a level just below the grid.

In all these distillation processes, steam is employed to carry the volatile odorous oil along with its vapours which are subsequently condensed to liquefy the oil and water vapours. Steam of different intensities is needed depending upon the volatility of the fragrant compounds in the plant material as well as on the thermo-stability of these components. There have been occasional improvements in the design of the still and the improved versions are employed for distilling lemongrass, palmarosa, vetiver, berries, geranium, patchouli, sandal and others. However, maintenance of low temperature and little water in direct contact with the material and uniform packing of raw material will lead to better yield of quality oil.

Equipments for distillation of oil

The main equipments required for carrying out essential oil distillation depends on the quantum of operation and method of operation. However, there are three main parts which, in different sizes form the base for all types of hydro-distillation. The retort, the condenser, the receiver and the fourth part is steam generator.

The retort or the tank commonly called is the primary container for the plant material wherein the water and steam contacts the plant material and vapourizes along with essential oil and moves on to condenser system. Steam containing the essential oil vapour leaves the still via a head, known as a gooseneck or through side outlet and passes to a condenser. Simple condensers consist of a metal coil in a tank of flowing cold water. There are different type of condensers like coil condenser and tubular condenser. The coiled condenser normally shortens space of condenser with the same condensation capacity. The tubular type of condensers is also simple in nature and inexpensive type consisting merely a series of long pipes offer considerable cooling surface area. Ideally the coil/tube of the condenser should be constructed from an inert material such as stainless steel in order to prevent the oil chemically reacting with mild steel. In many traditional stills the gooseneck and condenser coil were constructed from copper or brass that had been internally tinned to provide a reasonably inert surface. It is very important that the condensed steam leaving the condenser is thoroughly cooled. If it is still warm there will be a loss of essential oil.

Other specialized distillation like hydro diffusion, Cohobation and ratification in extraction of essential oil is employed depending upon the desired end product.

Hydro diffusion

This method is also similar type of steam distillation and only difference from the traditional steam distillation is here the steam is introduced from top onto the material instead from bottom. The condensation of the oil containing steam mixture occurs below the area in which plant material is held in place by a grill. The main advantage of this method is that less steam is used, shorter processing time and a higher oil yield.

Review

Cohobation in essential oil extraction

In the process of normal water distillation some of the constituents may get dissolved in water thus the distillate does not contain the complete constituents to be present *in-situ* of the essential oil[6]. Then the water is again distilled and added up in correct proportion to the essential oil distilled out to form the complete required essential oil. Eg. Rose oil.

When rose oil is extracted during water distillation, the one main constituent - phenyl ethyl alcohol dissolves into the water of the distillation still and does not form part of the essential oil that is so extracted. The oil so extracted is therefore not whole, and is deficient in this rose-smelling ingredient and in order to produce "complete" oil, the phenyl ethyl alcohol needs to be distilled from the water in which it dissolved and added back to the "incomplete oil".

Rectification in extraction of essential oils

In this process, when an essential oil contains any impurities, it can be purified by re-distillation either in steam or in vacuum and this purification by re-distillation is referred to as rectification.

Combination of water and steam distillation

This process is basically combination of normal water distillation and that of steam distillation. The raw material is immersed in water in a still, which has a heat source and to this live steam is fed and then normal condensation process is done.

OTHER METHODS OF EXTRACTION

Normal process of distillation has got its own limitations like action of steam on plant material may affect some of the important delicate constituents in the oil. To overcome this problem other methods of extraction like maceration, expression, enfleurage and extraction with volatile solvents are also employed in essential oil distillation.

Maceration

In maceration, the oil cells of the fragrant flowers are ruptured by immersion in hot fat or oil, which in turn absorbs the essential oils. Fat is separated from spent flowers and reused for absorbing fragrance from the next batch of fresh flowers. The fat retained by flowers is recovered by hydraulic pressing. The resultant perfumed pomade is frequently marketed as such but is often extracted with strong alcohol to yield extracts.

Expression

This method is employed when the essential oils are thermo-sensitive. In expression method there are two processes like the Ecuelle and sponge method are normally employed for isolating essential oils from lemon and orange peels. In Ecuelle process the whole lemons are rolled in hollow vessels covered inside with spikes to puncture oil cells, permitting the essential oil to ooze out into a collecting vessel for filtering to obtain clear oil. In *sponge* process, where the fruit is cut across the shorter axis and the peels are separated, steeped in water followed by hand pressing between sponges. The oil soaked sponges are squeezed to release the oil.

Enfleurage (Extraction with cold fat)

In this process, fragrance is made to absorb from flowers in contact with cold fats. This process is adopted for fragrant flowers like jasmine and tuberose, as solvents lack arresting the manifested fragrance. The fats should be saturated and odourless to prevent entrance of fat odours. The fat is thinly layered on both sides of a glass plate supported on a rectangular wooden frame. Fresh fragrant flowers are lightly layered on the fat coated chasses. Several chasses are placed one above the other sandwiching the flowers between two layers of fat. Spent flowers are removed (defleurage) and fresh charge is made. Reversing of the glass slab is called *patage*. Patage is done several times to obtain maximum perfume absorption. Furrows are created with combs to increase absorption surface. The process of defleurage and fresh charging are done every 24 hours.

Extraction with volatile solvents

This yields concretes and alcohol soluble absolutes having a near natural odour thereby making the enfleurage and maceration process obsolete. This process is presently employed regularly for the extraction of aromatic substances from flowers, leaves and mosses. The extraction is effected at room temperature using perfumery grade solvents like petroleum ether.

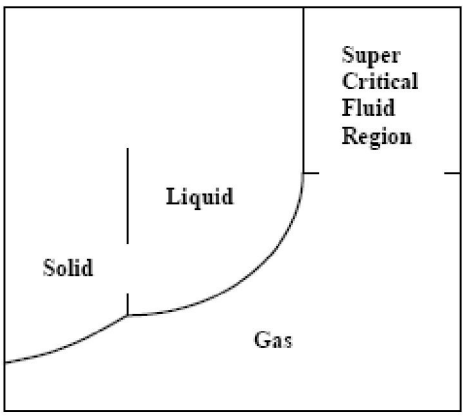
The solvent should be selected such that it should completely and quickly dissolve the odouriferous principles in the plant materials and should not dissolve inert principles of plant materials like waxes, pigments, and albuminous compounds and also be inert to flower oil constituents. The boiling point should not be too high or too low, it must be uniform. The solvent should be cheaper, noninflammable and immiscible with water.

MODERN METHODS OF EXTRACTION

Supercritical fluid extraction (SCFE)

This is an emerging and a versatile method to separate components that are susceptible to thermal degradation. This method is mainly employed for the extraction of flavours, fragrances and perfumes from a wide variety of natural products. This method of extraction is superior and faster than distillation. Higher diffusiveness and lower viscosities of supercritical fluids enable better penetration and faster equilibration. A supercritical fluid is any substance at a temperature and pressure above its thermodynamic critical point. It can diffuse through solids like a gas, and dissolve materials like a liquid. Carbon dioxide is the most commonly used solvent by virtue of its cheapness, non-toxicity, non-corrosiveness, non-flammability, easy to handle, needing mild processing conditions during extraction.

Essential oils have traditionally been derived using either steam or hydro distillation, or extracted using chemical solvents such as hexane or ethanol. The supercritical CO_2 extract and its process have many benefits over the traditional distillation processes. The CO_2 extraction process consists of pumping pressurized carbon dioxide into a chamber filled with plant ss



Emerging trends in distillation unit design

Innovative designs and technological advancement in the field of essential oil extraction has been emerging to achieve better performance in distillation and yield of the end product. In that recently, Microwave heating has occupied an incontestable place in analytical and laboratory level distillation. Micro wave accelerated extraction apparatus has been designed for qualitative and quantitative analysis of essential oils at the laboratory level and has been compared with the traditional Clevenger apparatus which has shown positive trends in terms of time and energy saving with better performance[8]. On the same lines better innovative commercial scale designs can add up to the future extraction systems in the field of essential oil distillation. Shock wave assisted distillation techniques have also shown positive trends in distillation of essential oil which can be the new generation modern technique in the field of essential oil distillation[1].

Basic parameters for design of essential oil distillation equipment

- Suitable technique for the distillation should be chosen on basis of oil boiling point and nature of material, as the heat content and temperature of steam can alter the distillation characteristics.
- Proper designing of tank, condenser or separators can lead to better yield of oils and minimize high capital investments.
- Suitable material to fabricate the still should be selected as some of the essential oils may be corrosive in nature.
- Proper control of injection rates and pressure in boiler operated units is necessary to optimize the temperature of extraction for maximal yield.

Few basic precautionary measures to get optimum performance

- Pre processing of raw material (crushing, powdering and soaking in water) may be done for some raw materials like roots and seeds to expose their oil cells to get better yield of essential oil, as such these materials will not yield essential oil easily if distilled in their natural state.
- Improper loading of the plant material may result in steam channeling causing incomplete distillation.

Review

- Efficient condensation system should adopt to avoid distillation loss.
- Sufficient distillation time should be given to complete the process as some of the high boiling fractions are not lost during the process.

Future needs

Cost effective, high yield, less water consuming, easy handling with better portability equipments are required for achieving the desired products in the field of essential oil distillation.

REFERENCES

[1] A.N.Arun Kumar, Y.B.Srinivas, G.Ravikumar, K.H.Shankaranarayana, K.S.Rao, G.Jagadeesh; A New Shock Wave Assisted Sandalwood Oil Extraction Technique. 24th International Symposium on Shockwaves Proc, Beijing China, July 2nd, **(2003)**.

[2] L.K.Doraiswamy, Ferreira, R.S.Sandra, M.Meireles, A.Angela, Nikolov, L.Zivko, Petenate, Ademir; The Journal of Supercritical Fluids, **14(3)**, 235-245 **(1999)**.

[3] Ernest Guenther, The Essential Oils, History-Origin in Plant Production-Analysis, D.Van Nosterand Company Inc, NewYork, **1**, **(1948)**.

[4] Elisa Maria Bittencourt Dutra de Sousa, Ana Paula Costa Câmara, Wallace Albuquerque Costa, Adriano Cezar J.Costa, Humberto Neves Maia Oliveira, Elisângela Lopes Galvão, Márcia Maria Ortiz Marques; Brazilian Archives of Biology and Technology, **48**, 231-236 **(2005)**.

[5] B.Rajendra, G.N.Gupta; Indian Perfumer, **8(2)**, 87-90 **(1964)**.

[6] S.K.Srivastava, M.C.Nigam; A review. Curr.Res. Med.Arom.Plants, **1(3)**, 147-155 **(1979)**.

[7] K.S.Theagarajan, V.V.Prabhu, G.Ravikumar; Ind.Perf., **37(2)**, 181**(1993)**.

[8] Weerachai Phutdhawong, Rungthip Kawaree, Samart Sanjaiya Waya Sengpracha, Duang Buddhasukh; Molecules, **12**, 868-877 **(2007)**.