Annexure III

A. Distillation of essential oil

Distillation accounts for the major share of essential oils being produced today. The choice of a particular process for the extraction of essential oil is generally dictated by the following considerations:

a) Sensitivity of the essential oils to the action of heat and water.

b) Volatility of the essential oil.

c) Water solubility of the essential oil.

As most of the essential oils of commerce are steam volatile, reasonably stable to action of heat and practically insoluble in water hence are suitable for processing by distillation.

1. Water or hydro distillation
2. Steam cum water distillation
3. Steam distillation

Water / Hydro distillation

Water or hydro distillation is one of the oldest and easiest methods being used for the extraction of essential oils. In this method the plant material is fully dipped in the water. The primitive 'Bhapka' method is based on this principle. In the Bhapka method the distillation still is made of copper. The still is fitted on brick furnace and the plant material is filled in the still and entirely covered with water to the top. Another copper vessel with a long neck is placed in a water tank or natural pond to serve as condenser. A bamboo pipe is used to connect the vapour line and mud is used to seal the various joints. The oil vapour along with steam is condensed in the copper vessel and is separated. These types of units are still being used for the preparation of 'Rooh' and ' Itr' of Gulab, Khus, Rajanigandha, Bela, Sewali phool, etc. In Hojai some of the agar distillers still continuing this method with little changes. This process suffers from serious drawbacks such as:

1. As the plant material near the bottom walls of the still comes in direct contact with the fire from furnace, there is a likelihood of its getting charred and thus imparting an objectionable odour (burning note) to the essential oil.
2. Prolong action of hot water can cause hydrolysis of some constituents of the essential oils such as ester, etc.
3. The process is slow and the distillation time is much longer thereby consuming more firewood / fuel.

Water cum Steam Distillation (Field Distillation Unit / Agar dag)

To remove the above drawbacks of water distillation the design has been improved where a perforated grid / net is introduced into the still just above the bottom. This type of unit is known as Field Distillation Unit (FDU) / Automatic Dag in case of agar oil distillation

(since manual stirring of the material is not done here). The main components of this process are:

i) Distillation still (usually made of stainless steel or food grade SS)
ii) Condenser (usually tubular multitube condenser)
iii) Oil separator / receiver
iv) Brick furnace

The still / tank is made up of mild steel / stainless steel with a perforated grid and is fitted directly on a brick furnace. The plant material is kept on the grid and water is filled below it. The tank is connected to the condenser through a vapour line. The condensate oil / vapour mixture is separated in the oil separator.

Due to their very simple construction, low cost and easy operation the field distillation units are extremely popular. The furnace is fuelled by spent agro-waste, firewood. These types of units are currently being used for the distillation of citronella, lemongrass and agarwood, etc. Incase agar distillation the distillate is cohobated manually that means returned back to the bottom (functioning as boiler).

Steam Distillation

The main components of steam distillation unit are:

1. Distillation tank with steam coil
2. Condenser (usually multi-tube tubular)
3. Oil separator or receiver
4. Boiler

Steam distillation exploits the twin action of heat and moisture from steam to break down the cell walls of the plant tissues to liberate the essential oil.

Steam is generated separately in a steam boiler and is passed through the distillation tank through a steam coil. The plant material is tightly packed above the perforated grid (false net). Steam along with oil vapours is condensed in the condenser and is separated in the oil receiver.

Capital cost of putting up steam distillation unit is higher and also a trained person is required for operation of the boiler. Steam distillation is preferred where a lot of area is under cultivation and more than one unit is installed. Also for distillation of high boiling oils such of roots / woods for example agarwood chips), patchouli, etc.

USE OF NATURAL GAS

In and around Sibsagar of Upper Assam natural gas supplied through underground pipeline is being used for commercial disstillation of essential oils like Agar oil. It has been observed that besides lowering the cost of distillation, the oil recovery is also higher in lesser time, This is due to the fact that, the fire can be regulated throughout which is not possible when firewood is used. The use of natural gas has become a boon for the distillers in those areas. More and more cottage level distillation units are being installed using natural gas as fuel. Use of natural gas will reduce the alarming air pollution being generated due to burning of wood or any other agricultural wastes in large quantities.

B. Clarification and storage of essential oils.

Usually the spoilage of an essential oil is attributed to some general reactions as oxidation, resinification, polymerization, hydrolysis of esters, and to interaction of functional groups. These processes seem to be activated by heat, by the presence of air (oxygen), of moisture and catalyzed by exposure to light and in some cases, possibly by metals. There is no doubt that oils with a high content of terpenes (all citrus oils, pine needle oils, oils of turpentine, juniper berry etc) are particularly prone to spoilage, due probably to oxidation, and especially resinification. Being unsaturated hydrocarbons, the terpenes absorb oxygen from the air. Light seems to be of lesser importance as a factor causing deterioration, than is moisture.

Essential oils containing a high percentage of esters (oil of bergamot, lavender, etc.) turn acid after improper storage, due to partial hydrolysis of esters. The aldehyde content of certain oils (lemongrass for example) gradually diminishes, yet much more slowly than if the isolated aldehyde (citral, in this case) were stored as such. Quite probably the essential oil contains also some natural antioxidants, yet unknown, which do a certain extent protect the aldehyde while it is contained in the oil. Fatty oils, with a few exceptions, are very prone to oxidation, but such spoilage can be prevented altogether by the addition of suitable antioxidants, such as hydroquinone or its monomethyl ether. Certain types of essential oils, especially those containing alcohols (geranium oil, for example), are quite stable and stand prolonged storage. Stills others, patchouli and vetiver, for instance, improve considerably on ageing; in fact they should be aged for a few years before being used in perfume compounds.

As a general rule, any essential oil should first be treated to remove metallic imurities, freed from moisture and clarified, and then be stored in well-filled, tightly closed containers, at low temperature and protected from light.

The small lot can be dehydrated quite readily by the addition of anhydrous sodium sulphate, by thoroughly shaking, standing for overnight or 6-8 hours and filtration. Calcium chloride must never be for dehydration as an essential oil, as this chemical is apt to form complex salts with certain alcohols. Larger commercial lots of oil are always easy to clarify. Some oils, such as vetiver, give a great deal of trouble. The simplest procedure is to add a sufficient amount of common salt to the lot, to stir the mixture for a while, and to let it stand until the supernatant oil has become clear and can be drawn off from the tank. The lower layer will be cloudy and needs to be filtered clear. If filtration through plain paper does not give clear oil, kieselguhr or specially prepared filtering clay should be placed into the filter. Care must be exercised in the selection of filtering medium as some media; activated carbon for example, may react chemically with certain constituents of the oil and affect its quality. Large quantities of oil should be filtered through filter presses, which are readily available. Centrifuging in high-speed centrifuges is an excellent means of clarifying essential oils. Not only moisture but also waxy material depositing after a certain period of storage, if possible at low temperature in a freezing room, can thus be eliminated.

Some lots of essential oils, especially those with a high content of phenols (clove, bay, thyme, origanum, etc) arrive from the producing fields often in a crude form and dark

coloured, due to the presence of metallic impurities. Such lot must be decolourized before they can be placed at the disposal of the consumer. This dark color may be removed by the formation of complex salts with certain organic acids. For this purpose sufficient powdered tartaric acid is added to the oil, the mixture stirred for some time and permitted to settle. The supernatant clear oil can finally be drawn off, while the lower layer has to be filtered until clear. If the treatment with solid tartaric acid does not give satisfactory results, a concentrated aqueous solution of the acid is added to the oil. After thoroughly stirring the mixture is allowed to stand until the two liquid layers separate clearly. The upper part of the oil layer should then be sufficiently clear to be drawn off, while the lower layer

and especially the intermediary layer, need further treatment by clarification and filtration. Here again high-speed centrifuge are of great help. In case where the colour cannot be eliminated by treatment with organic acids, the oil will have to be clarified by reinstallation or rectification.
If the oil quantity is less they can be easily stored in bottles of hard and dark coloured glass. For larger quantities of oil stainless steel or aluminum containers should be preferred. During storage oil should be filled upto the brim and containers should be kept in shaded / cool areas away from direct heat and sunlight. A layer of carbon dioxide or nitrogen gas blown into container before it is sealed will replace the layer of air above the oil and thereby assure added protection against oxidation.

Processing of medicinal plants

Processing of medicinal plants depends on the activity and utilization of the oils or desired compounds found in their leaves, barks or roots.

Production of medicinal extracts and isolates can be broadly classified into two parts mainly on the basis of their use. First those in which the extracted reactive component, active ingredient or phyto pharmaceuticals i.e., the alkaloids are directly used in modern medicine system that is Allopathic on quantified basis. For extraction of these kinds we generally use solvent extraction method.

Second classification is mainly based for those roots / barks /stems, etc where they are directly used in traditional medicinal system such as Ayurveda, Homeopathy, Sidha, Unani, etc in the form of paste, powder, water / honey extracts etc.

The main steps involved in processing of medicinal plants generally include drying, size reduction, sieving, production of aqueous extracts, solvent extraction, column chromatography, spray drying and standardization.

Quality Assessment

Every essential oil is basically a mixture of different components / compounds. The percentage of these constituents in the oil plays an important part in determining its quality. The apparatus by which the fractions and their percentage are determined is Gas Liquid Chromatographic unit (GLC). Most of the oils being sold in the market today are on the basis of GLC report. NEDFi R&D Centre has this facilities available for growers / processors / traders, etc. For medicinal extracts, TLC (Thin Layer Chromatography) and HPLC (High Pressure Liquid Chromatography) techniques are being used.