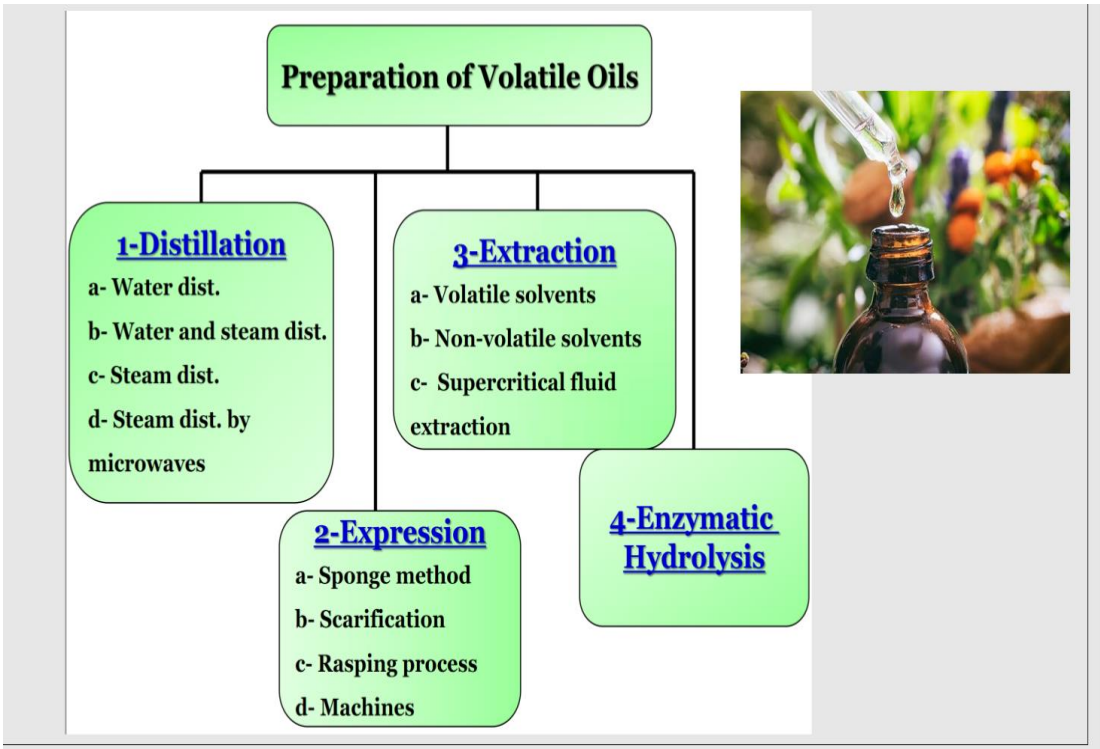
Tikrit University
College of pharmacy
Pharmacognosy lab.4

Extraction methods of volatile oil



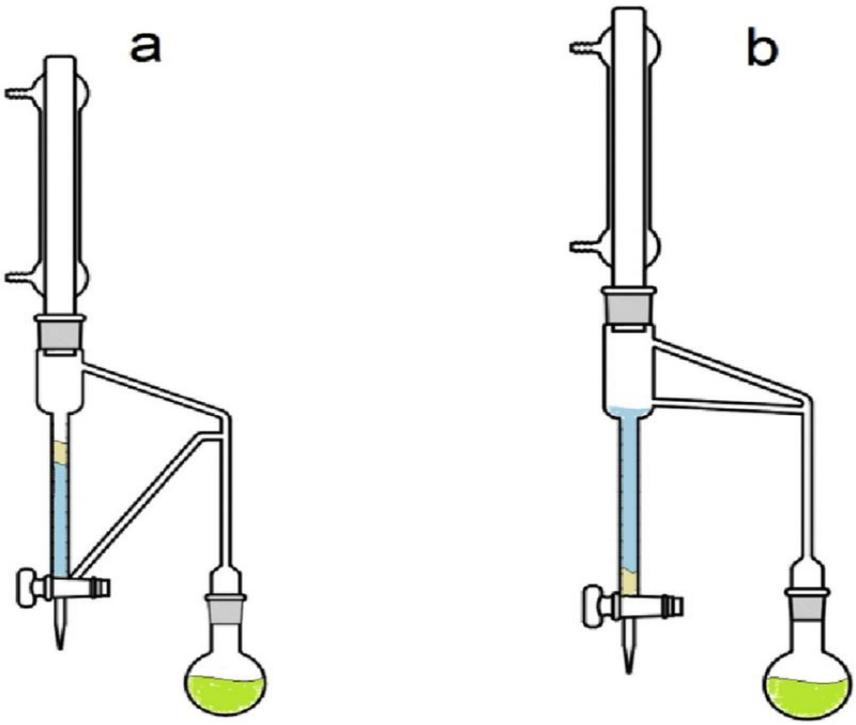
1- Distillation methods:

a) Water distillation (hydrodistillation):

1-The dried plant material is immersed in water saturated with NaCl.

2- Water is heated until boiling, and then the steam together with the V.O are condensed and received as two layers, one for water and the other for the V.O.

3- Cool and separate.



Important notes:

1- Clevenger apparatus is used in the water distillation method. It is available both for oils lighter than water and oils heavier than water.

2- Water distillation is suitable only for thermally stable V.O. Under the drastic conditions of distillation, unstable V.O. will undergo degradation.

3- This method is used for dried plant material, which keep their odour for long time (oils are secreted from internal secretory structures). Thus, preparation of the volatile oil is under drastic conditions (boiling).

4- NaCl is added to minimize the solubility of the distilled oils in water.

Disadvantages:

1- Some compounds may be subjected to hydrolysis, e.g. esters.

2- Complete exhaustion of the plant material is difficult as this process is insufficient for high boiling point compounds.

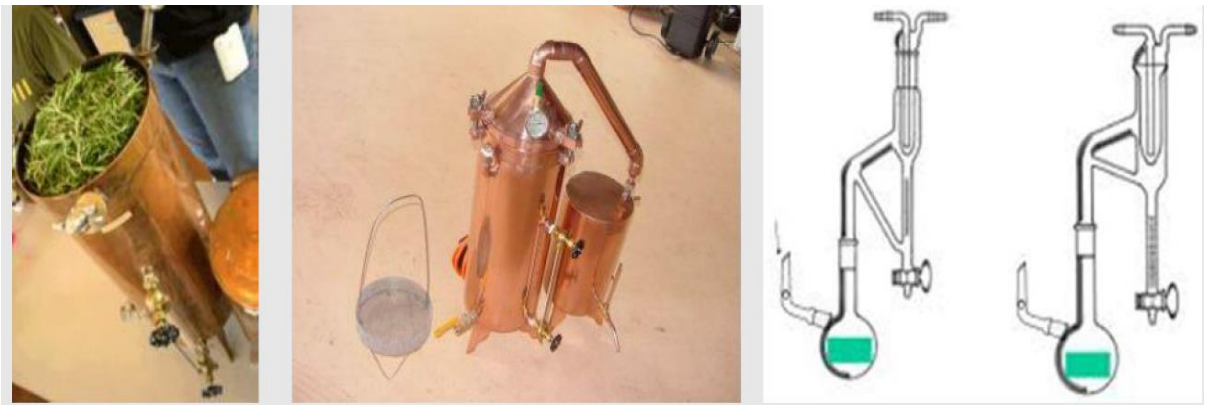
b) Steam distillation:

1- It is used only for fresh plant material (especially for those containing oils in external secretory structures).

2- The plant material is cut and transferred to a distilling chamber (suspended in a wire basket or perforated trays).

3- Steam is forced through the plant material and the oil is obtained as before.

4- Mostly used in the large scale production of V.O.



c) Water and steam distillation:

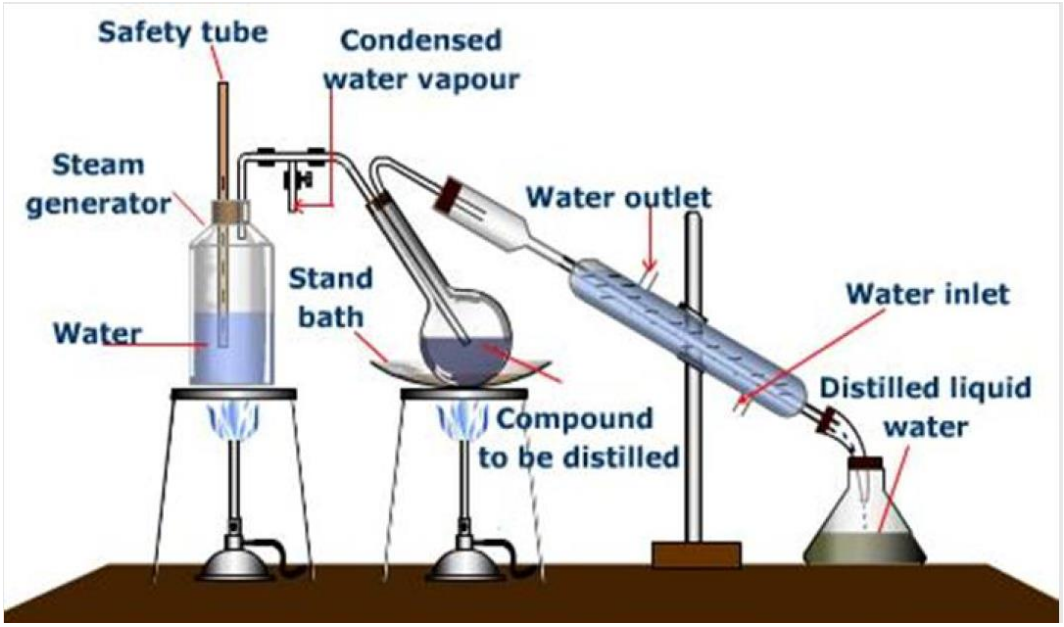
1- The plant material (fresh or dried) is immersed in water.

2- Water is heated and at the same time, the steam generated elsewhere is piped into the container containing the macerated material.

3- The steam is condensed and the oily layer is separated as before.

4- This method provides some sort of heat control to prevent overheating and decomposition of V.O.

5- Useful in case of hard plant material like seeds or barks.



d) Steam distillation by microwaves:

1- A new technology that include steam distillation by microwaves under vacuum.

2- The plant is heated selectively by microwave radiation in a chamber inside which the pressure is reduced sequentially. The V.O is distilled away with the water vapours from the plant itself (fresh plants do not require the addition of water).

3- This method is fast, consumes little energy, and yields oils of high quality.

General notes on distillation methods:

◦ Sample pretreatment is necessary (cutting, crushing, etc.).

◦ Using very high temperatures will not only affect the oil constituents (hydrolysis or decomposition) but may also affect the other plant components.

◦ Complete distillation is essential to get all the V.O. constituents.

◦ Iron containers should not be used for distillation processes of oxygenated compounds because the oil will darken in colour. Therefore, stainless steel or copper lined with tin containers should be used.
◦ Oil recovery: the collected distilled water and V.O are received in the oil separator, where the oil can be easily separated. However, the water will keep some of the obtained volatile oils either in solution or suspension, leading to loss of some volatile oils; therefore, Cohobation is recommended, where the water can be freed from the oil by saturation with NaCl or by extraction with an organic solvent.

2- Expression methods:

- Before the discovery of distillation, all essential oils were extracted by pressing.
- These methods are used for preparation of V.O occurring in the rind of fruits, e.g. peels of orange, lemon, bergamot, etc. They are also the most suitable method for sensitive oils, such as that in the rind of Citrus fruits.

3- Extraction methods:

◦ Volatile oils which decompose by heat or present in trace amounts in their plant materials are usually prepared by this process. Extraction methods include:

a) Extraction with volatile organic solvents:
◦ 1- The plant material is extracted with certain organic solvents of low polarities, such as hexane, petroleum ether or benzene in a Soxhlet apparatus.
◦ 2- The resulting extract is evaporated under reduced pressure leaving a residue of V.O and other less polar compounds of the plant, e.g. pigments and waxes. The obtained residue is known as floral concrete.

◦ 3- The absolute oil (or the absolutes) is obtained by dissolving the floral concretes in absolute alcohol thus precipitating the albuminous materials, waxes, and pigments, followed by concentrating the alcoholic solution. The absolute is the most pure and expensive form of the oil.

◦ Advantages of extraction with volatile solvents over distillation:

a- Much lower temperature is used (not more than 50 °C).

b- The prepared oil keeps its natural odour.

b) Extraction with non-volatile solvents:

b-1) Maceration:

The plant materials, usually fragrant flowers, are allowed to remain in contact with inodorous fixed oils or fats, which absorb the odorous principles. The total oily portion is then separated by filtration.

b-2) Digestion:

This process is similar to maceration but moderate heat is applied to aid extraction.

b-3) Enfleurage:

◦ - This process was once used for extraction of volatile oils of delicate flowers, e.g. Jasmine.

◦ - Two thin layers of inodorous fat (beef tallow or lard) are spread and flowers are then spread lightly between them.

◦ - After complete extraction, the flowers are removed (defleurage) and replaced with new flowers.

◦ - With prolonged contact, the fat layers become saturated with the volatile oil of the flowers.

◦ - These fatty layers, called pomade, are extracted with alcohol (stirred with alcohol that dissolves the oil leaving the fat).

◦ - The oil is separated by fractional distillation from alcohol. The resulting oil, which is free from both fat and alcohol, is called absolute or enfleurage.

b-4) Pneumatic method:

◦ The same principle of enfleurage method but includes passing a current of warm air through the flowers. The air loaded with the oil is passed through a spray of melted fat in which the oil is adsorbed and separated as before.

c) Extraction with a supercritical fluid (supercritical gas extraction):

◦ At the supercritical pressure & temperature, gases have two important characters: flow ability like a gas & extraction ability like a solvent. Thus, supercritical gases/fluids can defuse through plant tissues and extract essential oils.

◦ Carbon dioxide (CO_2) is the most used supercritical fluid. Extraction conditions for supercritical CO_2 are above the critical temperature of 31 °C and critical pressure of 74 bar.