

# Isolation, Identification and Analysis of Menthol

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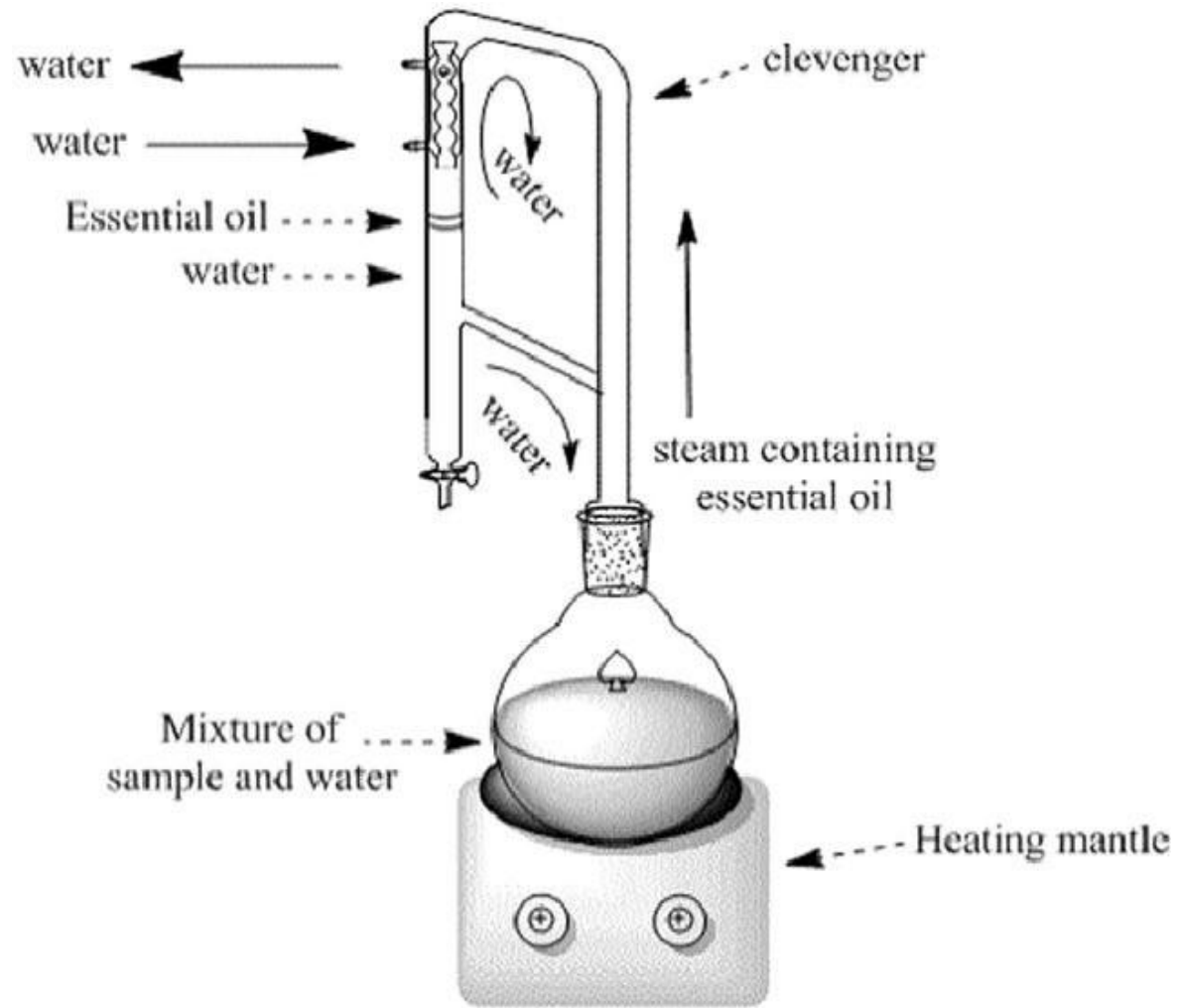
# Menthol

- Biological source: Menthol a monoterpene alcohol obtained from oils of *Mentha piperita* var. *Vulgaris* (Black peppermint) & *Mentha piperita* var. *officinalis* (White peppermint), family- Labiate. *Mentha* species contains about 1-3% of volatile oils.
- Isolation
- Menthol is isolated by 2 methods
- Method 1: Hydro-distillation
- Method 2: Steam distillation

# Hydro-distillation

- Required quantity of coarse powder leaves of *Mentha piperita*.
- The peppermint oil is extracted by hydrodistillation method by using clevenger apparatus.
- The oil is separated from water and allow to cooling. After cooling crystals of (-) menthol will separate out.
- The crystals are collected by centrifugation and re-crystallized the menthol by acetone or any other low boiling point solvent

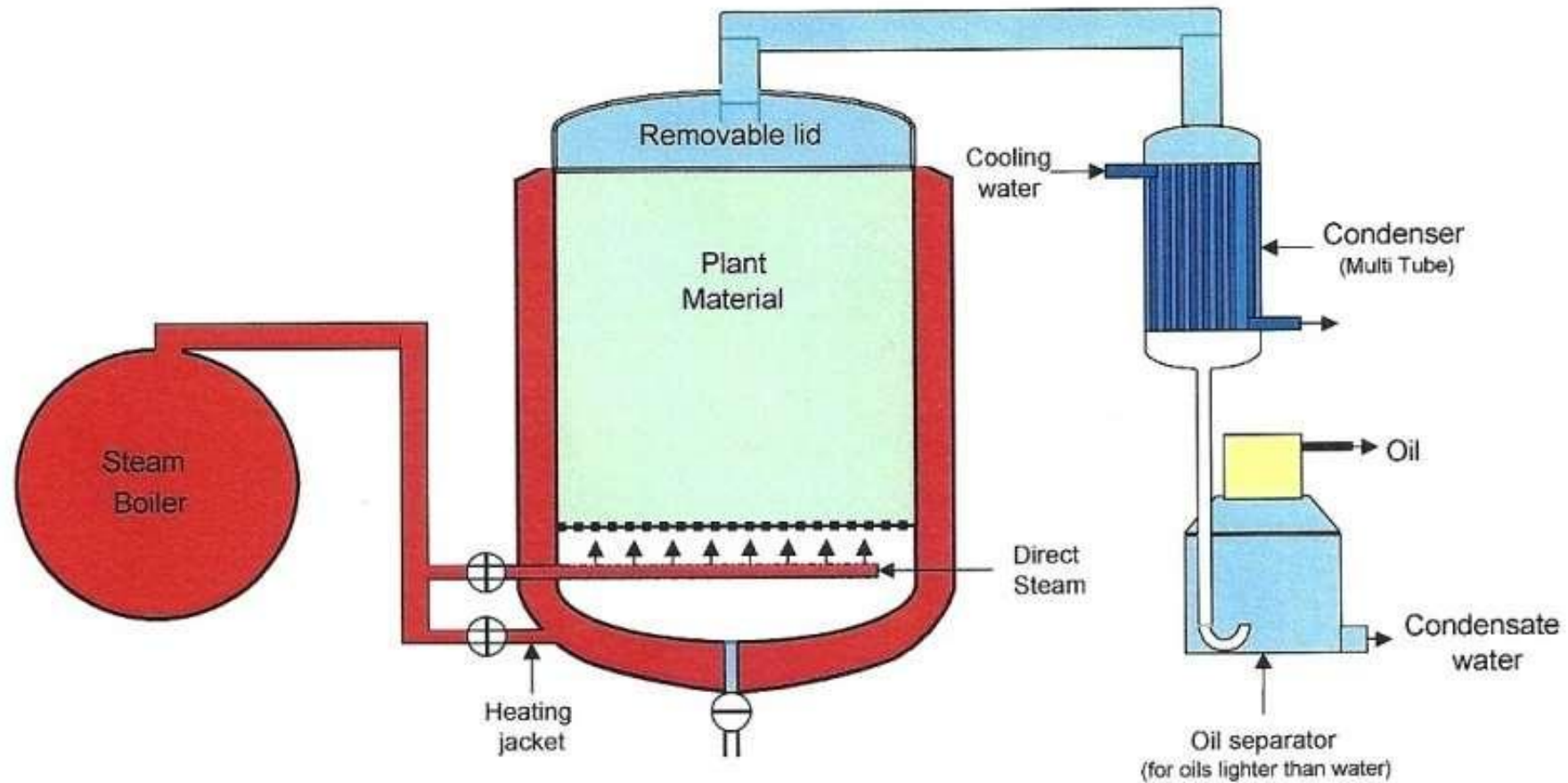
# Hydro-distillation by Clevenger apparatus system



## 2. Steam distillation

- Required quantity of air dried mentha plant is charged into stainless steel still having perforated bottom.
- The steam under pressure is generated with the help boiler and steam is passed through the drug.
- The mixture vapour (water and volatile oil) are passed through the condenser where vapour is cooled and back to liquid form.
- The mentha oil is collected in separating can.
- Mentha oil is floated on top of the water due to lighter than water.
- The oil is then decanted and filtered.
- The oil is allowed to cool; crystal of menthol will separate out.

## 2. Steam distillation



# 3. Method of Isolation

- Peppermint volatile oil- separated by steam & water distillation.
- To make oil, free of moisture- it is passed through a bed of anhydrous  $\text{Na}_2\text{SO}_4$ .
- The moisture free oil is frozen to  $-60^\circ\text{C}$  for 7days by packing in tightly sealed plastic container.
- Menthol in oil separates as flaky crystals, separated by filtration.
- Mother liq. Containing some menthol along with other terpenes.
- The mother liq is treated with boric acid & further boiled for 3hr, followed by distillation for removal of menthone.
- The Borate of menthol subjected to saponification with 50ml of NaOH by heating reflux for 1hr
- Resultant sol. is allowed to cool for separation of remaining menthol crystals and mixed, dried in dessicator.

# Properties:

- Appearance : White crystalline substances, which is solid at room temperature and melts slightly above (m.p. 41 to 43°C).
- Odor : Characteristic and pleasant
- Taste : Pungent followed by cooling sensation
- Solubility : Soluble in 70% alcohol, ether and chloroform, insoluble in water
- **Identification by chemical test:**
- Few drops of sample is mixed with 5ml of nitric acid and heated on water bath. Blue color is developed within 5 minutes, after some time it becomes yellow which indicate the presence of menthol.
- Small qty of menthol in TT + equal qty of thymol or camphor → liquifaction of contents in TT → presence of Menthol
- Crystals of Menthol in watch glass+ heat on water bath → material evaporates



## **Analysis by TLC**

- Sample preparation : 1mg of Menthol is dissolved 1ml of methanol
- Standard sample : Menthol
- Stationary phase : Silica gel 60 F 254
- Mobile phase : Pure Chloroform , hexane:EtOAc::8:2
- Detecting agent :1% vanillin – sulphuric acid reagent and heat the plate at 110°C for 10 minutes
- Anisaldehyde –sulphuric acid reagent, heat at 115°C – 10min.
- RF Value : 0.34, 0.48-0.62

### **Utilization:**

- It is used as in various dosage forms for its cooling sensation, flavoring property,
- carminative, antispasmodic and antipruritic
- **Storage condition:**
- It should be store in well closed and air-tight containers protected from light and in cool place.