Lab N °07 : Extraction of menthol contained in mint leaves

Objectives

The main objective is to extract the volatile aromatic chemicals from the mint leaves or other plant components.

Introduction

Mint is one of the most well-known medicinal plants. Archaeologists have found mint leaves in Egyptian tombs. Its use is documented among the Greeks and Romans for pain relief or to purge the sick. Falling into obscurity in the West, it only returned to traditional pharmacopeia in the 18th century. Since then, it has been one of the first plants to be intensively used by the pharmaceutical industry. Menthol has thus become a classic item in pharmacy displays. Mint is also found in a large number of candies, syrups, or as a flavor intended to enhance the taste of certain medications.

Menthol is a naturally occurring compound found in mint plants, particularly peppermint and spearmint. Its chemical formula is $\boldsymbol{C_{10}H_{20}O}$, and it belongs to the class of organic compounds known as terpenoids. Menthol is a cyclic alcohol with a characteristic minty odor and taste.

Steam distillation is the most often used technique among them any extraction techniques. The mint plant material is heated using steam, which causes the essential oil to vaporize. The oil is then separated from the condensed water because of the vapor being condensed.

Medicinal properties of mint include:

- Digestive disorders: Effective for constipation or diarrhea.
- Urinary issues: Diuretic effect.
- Cough and cold: Soothing.
- Pain relief: Joint pain, muscle pain, and headaches.
- Respiratory problems: Effective against conditions similar to bronchitis.
- Skin conditions: Relieves pain from insect and animal bites and prevents the formation of cracks.

To obtain one liter of peppermint essential oil, 400 kg of peppermint plant is required. The essential oil extracted from American peppermint primarily contains menthol (40%), menthone (20%), neomenthol (4%), and cineole (4%).

Materials & Equipment

Materials	Equipment
Mint Leaves Diethyl ether Distilled water	Hydro distillation setup 100 mL graduated cylinder Separatory funnel Erlenmeyer flask Spatula

Procedure

Hydrodistillation or steam distillation

1- Place 100 g of mint leaves in a 500 mL flask with 300 mL of distilled water.

Under the effect of heat, the cells containing the aromatic substances burst (decoction). Since these substances are volatile, they can be recovered through hydrodistillation (or steam distillation).

2- Heat the mixture until Recovering 100 mL of distillate.

The low content of essential oil in mint and the partial solubility of this oil in water **do not allow** the observation of **two phases**.

Extraction in a separatory funnel

1- Transfer the distillate into 250 mL separatory funnel.
2- Add 10 mL of diethyl ether to extract the essential oil from water.
3- *Salting out:* Add approximately 2 g of sodium chloride (NaCl salt) to facilitate the separation of the essential oil by *saturating* the lower aqueous phase with salt. This
4- vigorously agitating until the turbidity of the aqueous solution disappears
5- After separating the two phases, discard the aqueous phase and recover the ethereal (organic) phase containing the mint essential oil.

Questions

1. What is essential oil?
2. Draw the setup used for extracting orange peels and name all its parts.
3. What are the main steps performed for the essential oil extraction?
4. Specify the role the 3rd step.
5. Calculate the yield of the prepared extract.

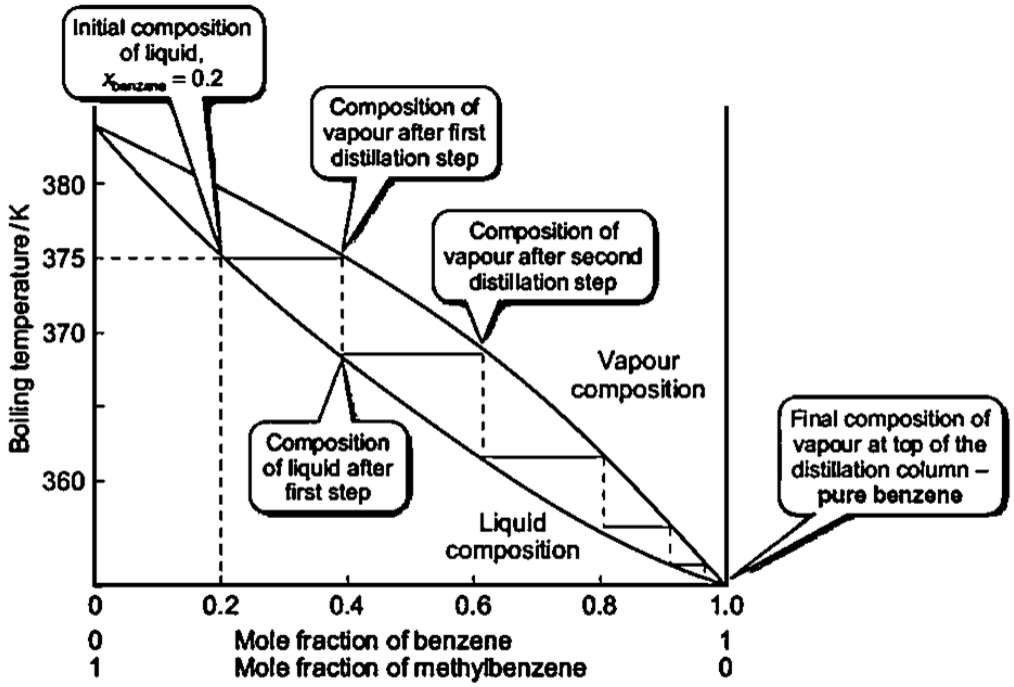
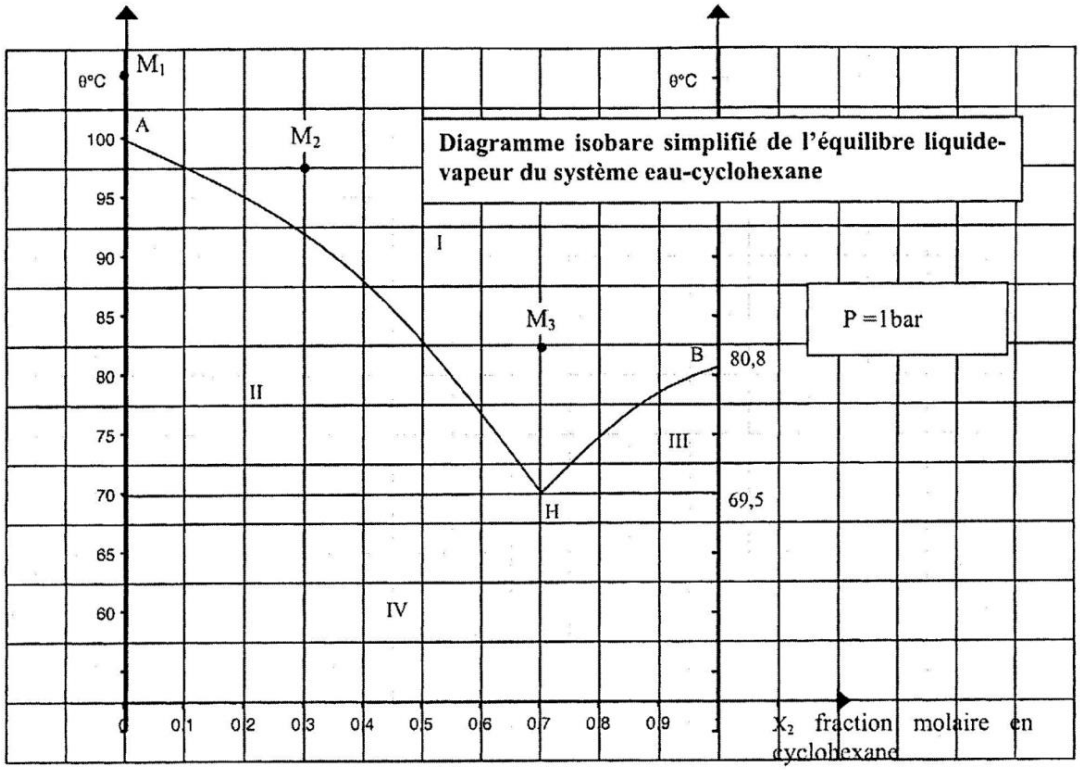


Figure 17.29 Fractional distillation can completely separate benzene and methylbenzene. In effect, a large number of successive distillations occurs as the vapour passes up the column.



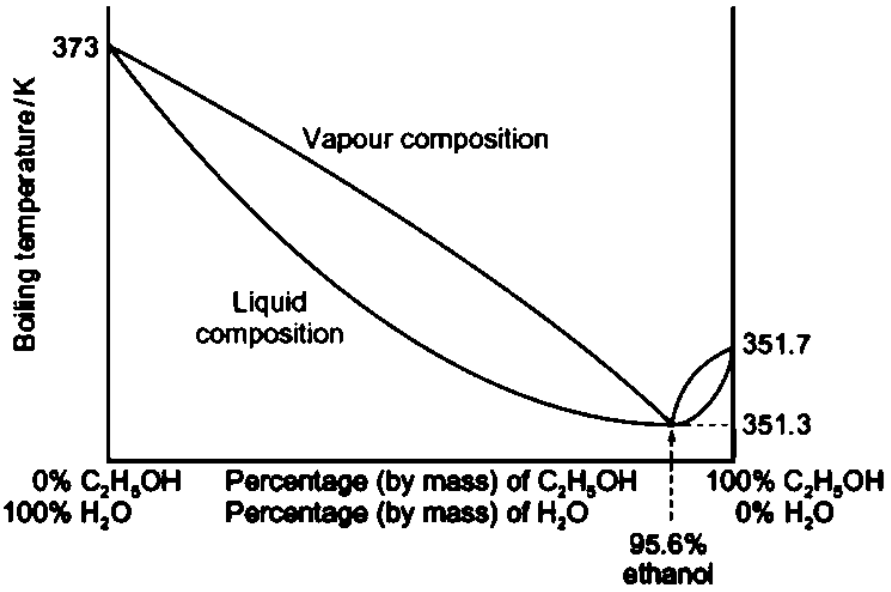


Figure 17.30 Ethanol and water form a minimum boiling azeotrope. These data refer to a pressure of 1 atm.

Salting out is a technique used in the extraction of essential oils to separate them from water or other aqueous solutions. It relies on the principle that adding a high concentration of salt to the aqueous phase can decrease the solubility of the essential oil in water, causing it to separate and form a distinct layer that can be easily collected.

Here's how salting out works in essential oil extraction:

Preparation: After performing the initial extraction (such as hydrodistillation or steam distillation), the essential oil is usually mixed with water or remains dissolved in the aqueous solution.

Salting out: A large amount of salt (usually sodium chloride, NaCl) is added to the solution. The salt disrupts the water's ability to solvate the essential oil molecules, causing them to aggregate and coalesce into larger droplets.

Phase separation: As a result of salting out, the essential oil forms a separate layer on top of the aqueous solution. This is because the solubility of the essential oil in the aqueous phase is reduced due to the presence of the salt.

Collection: The separated essential oil layer can then be easily collected using a separatory funnel or other appropriate extraction apparatus. The aqueous phase, containing water and some dissolved salts, is discarded.

Salting out is a simple and effective method for extracting essential oils from aqueous solutions, and it is commonly used in laboratory-scale extractions as well as in industrial processes.