Chapter 5 Process of extracting essential oils

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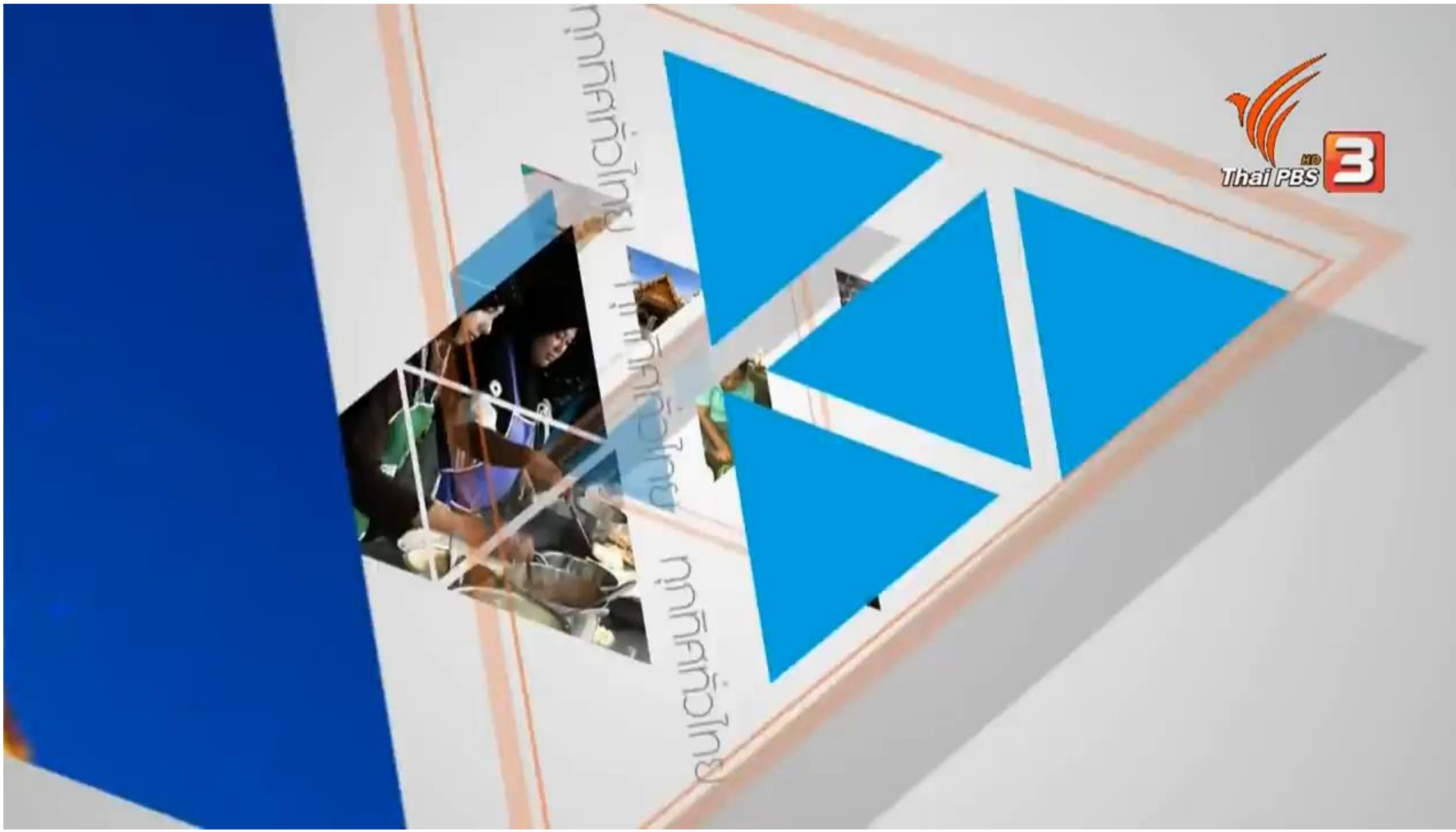
Topics

- Classification of raw materials used in the extraction of essential oils.
- Method for extracting essential oils from raw materials
- Essential Oil Quality Check

Classification of raw materials used in the extraction of essential oils.

Aarts of plants that contain essential oils	Essential oil samples
Flower	Rose, jasmine, orange blossom, ylang-ylang, cloves
Roots	ginger, ginger, vetiver
Fruit and seed	Coriander, cumin, cardamom, nutmeg
Leaf	Lime, citronella, lemongrass, eucalyptus
wood/bark/trunk/underground trunk	Cinnamon, pine, aloes, moonshine, ginger, galangal
Resin (used in incense or incense)	frankincense, various fragrant resins
fruit peel	Orange, lime, bergamot

Extraction form Lime Peel



Method for extracting essential oils from raw materials

The division of plants with essential oils into 3 groups.

1. Groups that have a lot of oil, such as various orange peels It can be clearly seen as an essential oil storage cell with the naked eye. When gently squeezed, oil comes out. and fragrant
2. Groups with medium oil, such as lemongrass, kaffir lime leaves, basil, basil, this group will not smell good when walking through this group. but must be crushed and smell good
3. Groups with little oil, such as flowers, jasmine, ylang-ylang, tuberose, which will smell far, soft, but when crushed, it will smell green. no fragrance

Method for extracting essential oils from raw materials

Essential oils must go through a process called "extraction" which can be done in 5 ways:

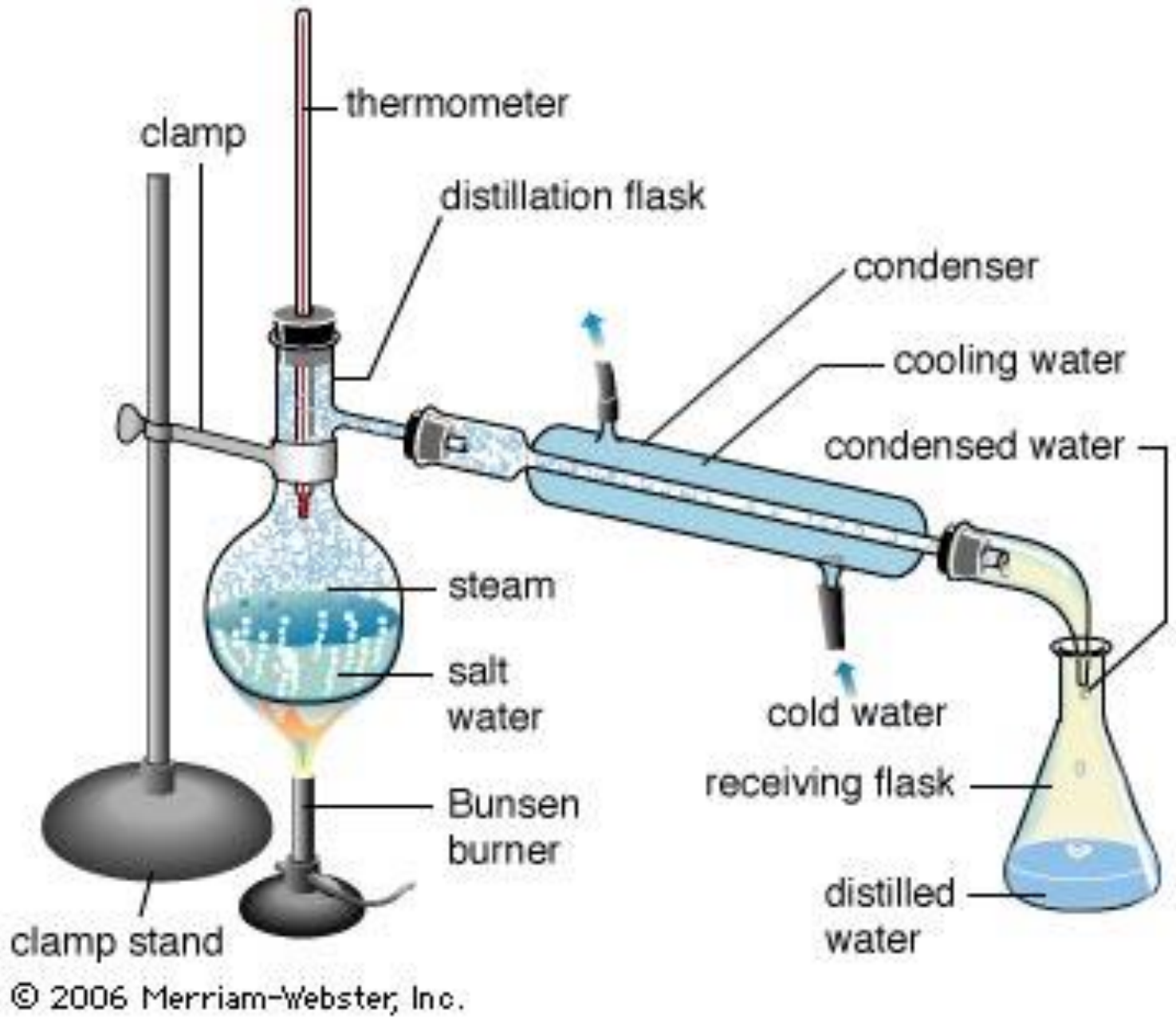
1. Distillation
2. Extraction by animal fat
3. Solvent extraction
4. Squeezing
5. Liquid Carbon Dioxide Extraction

Distillation

- It is a widely used method. Because it is an economical method and can be used to separate almost all essential oils.
- The important things to control in distillation are timing and temperature. because it will affect the quality and smell of the oil obtained
- The oil extracted by this method is Plai oil, Lemongrass oil.
- Distillation can be divided into 3 methods:
  1. Water distillation / hydrodistillation
  2. Water and steam distillation
  3. Steam distillation

Water distillation/ Hydro distillation

- It is commonly used with plants whose chemical composition does not decompose when heated.
- By bringing plants to be distilled into the distillation pot. then add water until it completely covers the plants boil until boiling water When boiling water evaporates into steam The steam will help release the essential oils contained in the plant tissues. when passing through the condenser The steam and essential oil vapor condense into a liquid. Water and essential oils are separated from each other.
- The disadvantage of this method is that in the case of distilling large quantities of plants, The heat given to the crucible is not uniform throughout the crucible. Cause burning or decomposition of certain elements, changing the aroma of essential oils. or may have the smell of the container attached
- For refining small amounts of plants in the laboratory we can do Using a distillation set made of glass called a Clevenger distillation set.


thermometer
clamp
distillation flask
condenser
cooling water
condensed water
steam
salt water
cold water
receiving flask
Bunsen burner
distilled water
clamp stand
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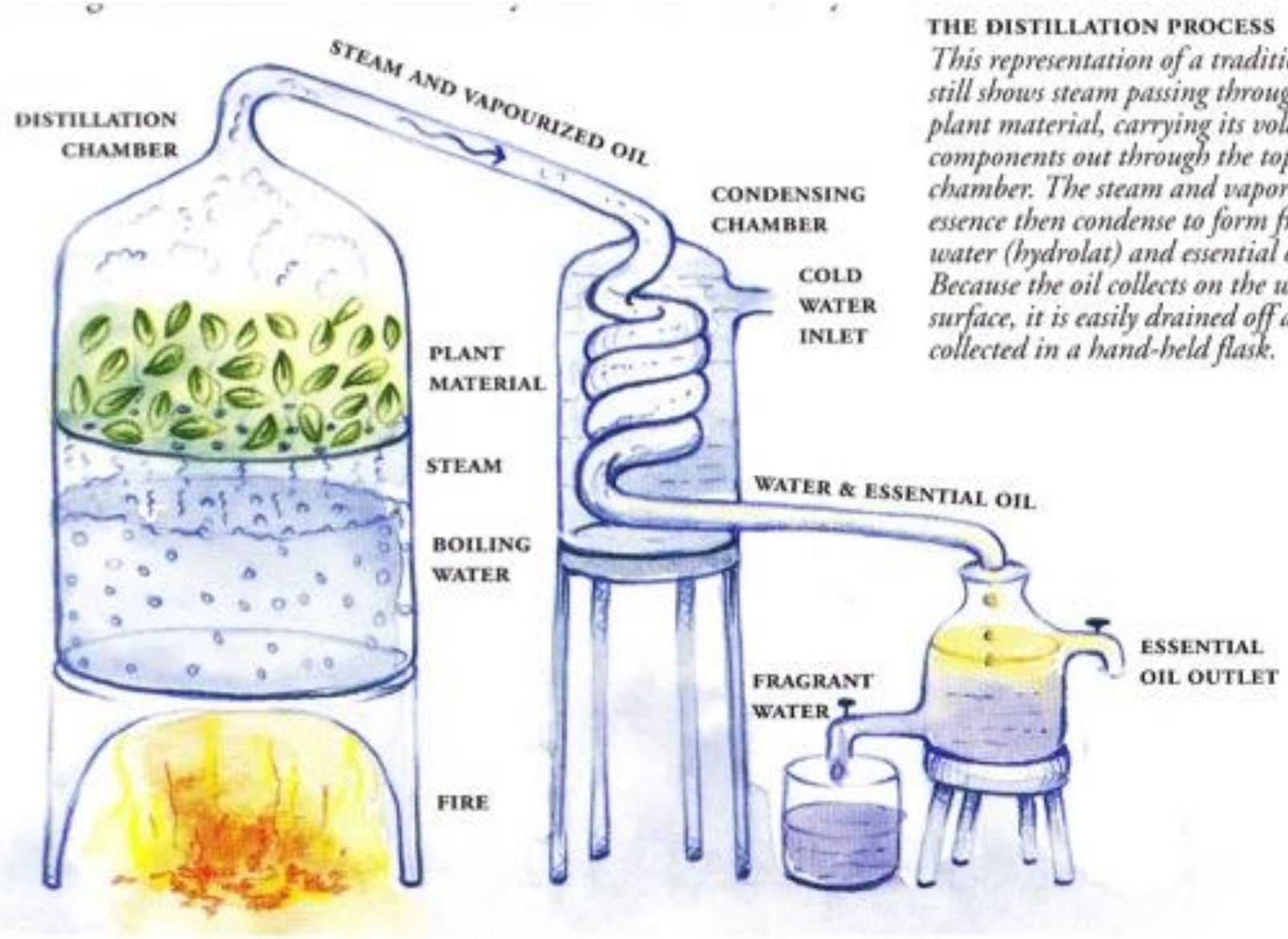
Water and steam distillation

- It is commonly used with plants whose chemical constituents decompose when exposed to direct heat.
- The plants to be distilled are placed on a sieve above the boiler. Heat until the water boils into steam. The steam will bring the essential oils and condense them back into water and essential oils.
- Distillation in this method may be called Wet steam. Plants used in this distillation are of better quality than the first method.

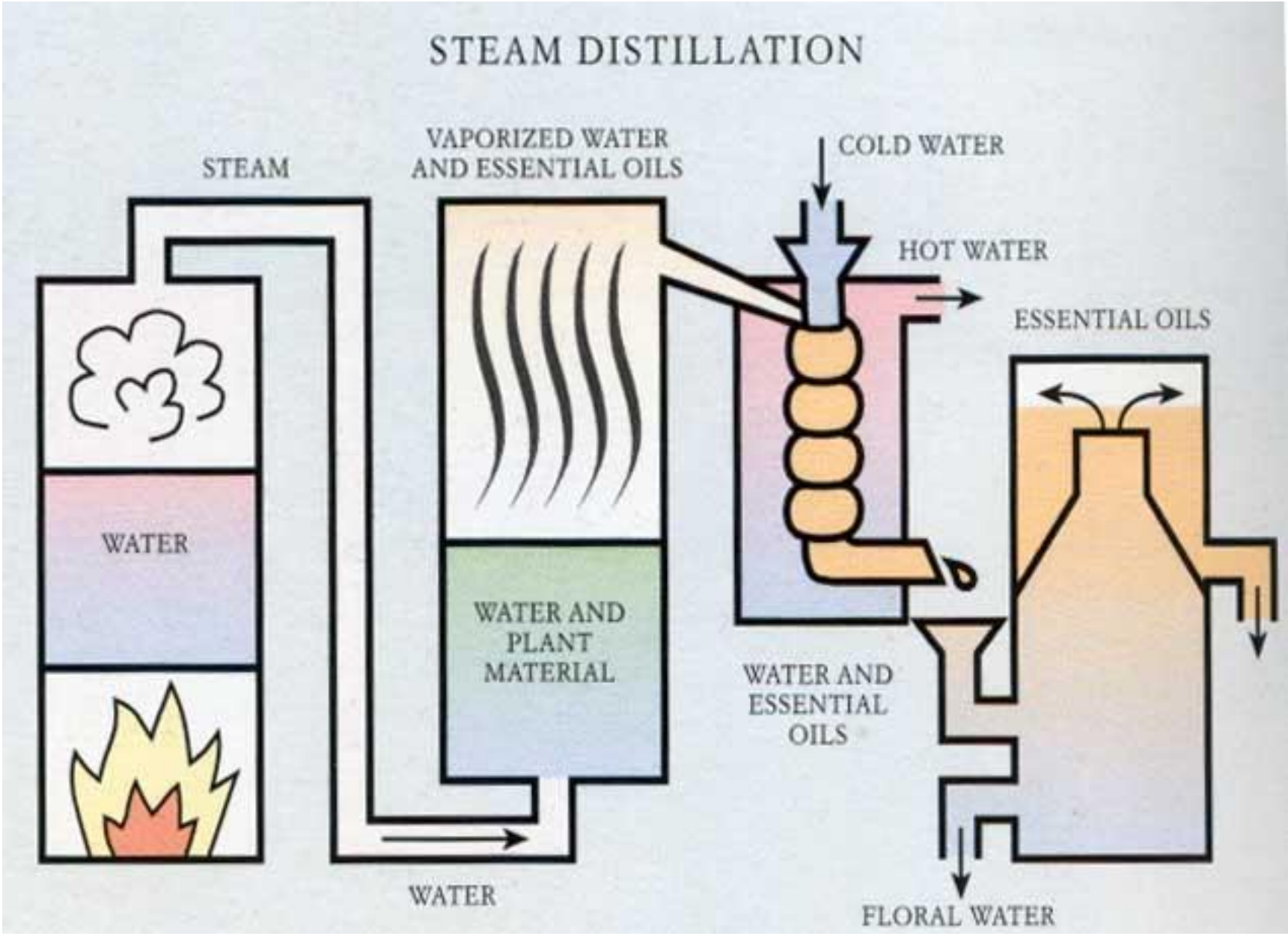


THE DISTILLATION PROCESS

This representation of a traditional still shows steam passing through plant material, carrying its volatile components out through the top of the chamber. The steam and vaporized essence then condense to form fragrant water (hydrolat) and essential oil. Because the oil collects on the water's surface, it is easily drained off and collected in a hand-held flask.

Steam distillation

- By bringing the plant to be distilled on a grid above the distillation pot to pass heat from steam.
- Water will quickly evaporate essential oils in plants. The advantage of this method is Short refining time and oil
- The quality and quantity of essential oils were higher than the first two methods.
- Unsuitable plants for distillation in this way are: Thin plant parts, such as rose petals, should be used with a fat extraction method.


STEAM DISTILLATION
STEAM
VAPORIZED WATER AND ESSENTIAL OILS
COLD WATER
HOT WATER
ESSENTIAL OILS
WATER
WATER AND PLANT MATERIAL
WATER AND ESSENTIAL OILS
WATER
FLORAL WATER

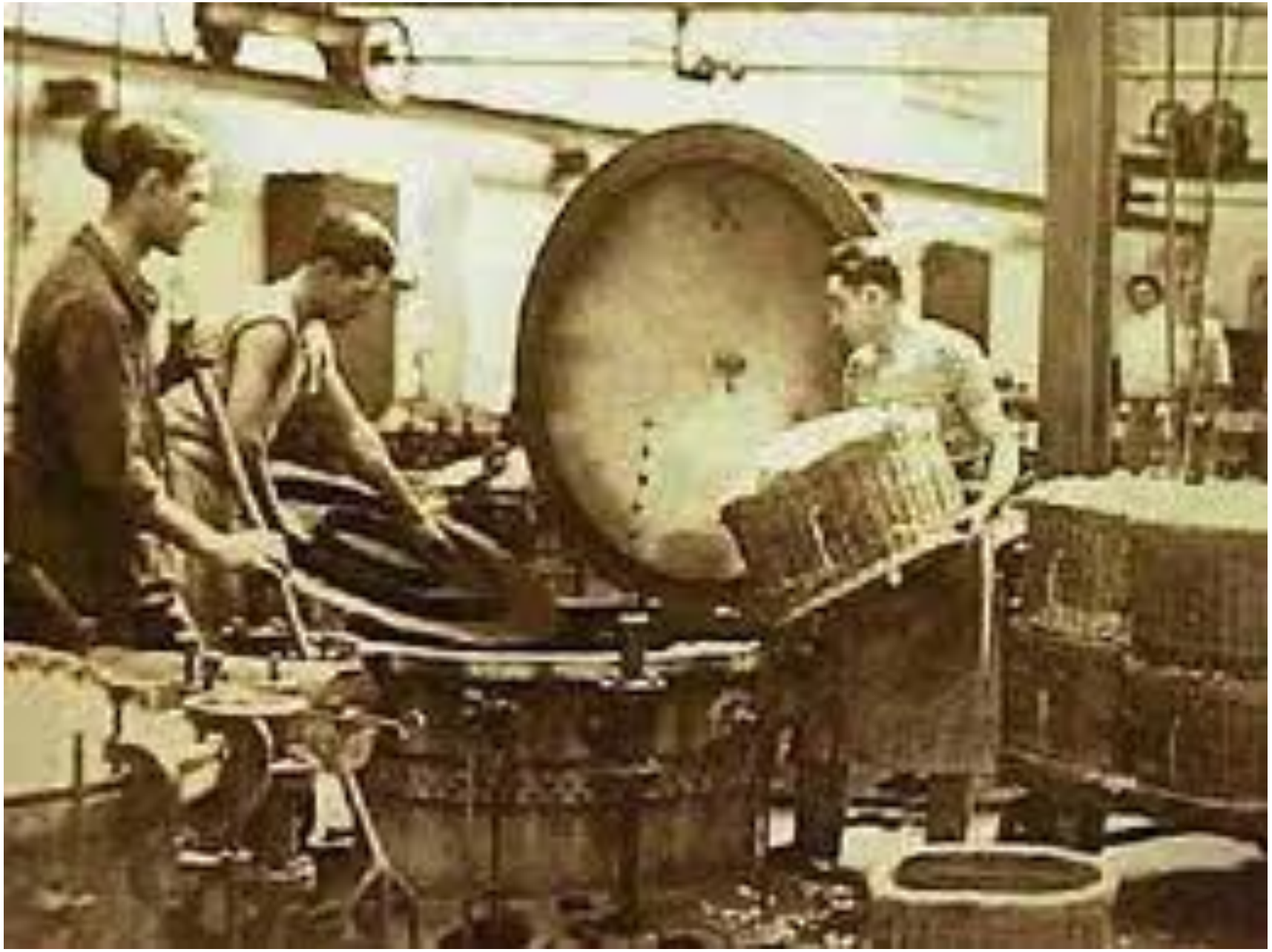
Extraction of *Michelia alba flower*

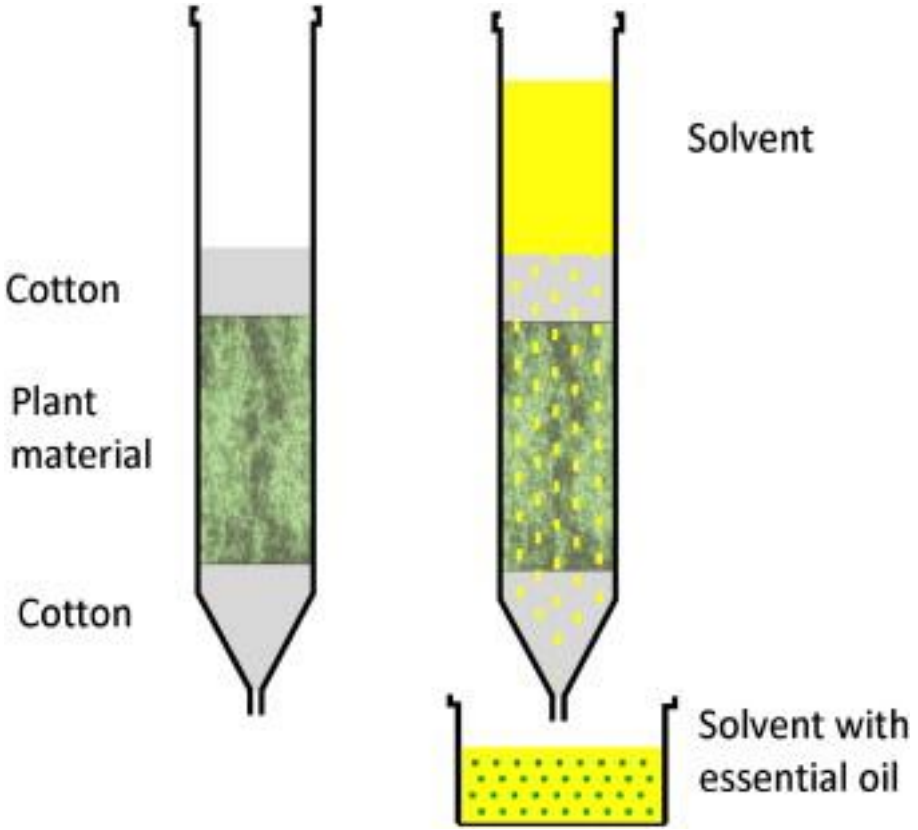
Extraction by animal fat

- Called as Enfleurage
- This method is used for essential oils that volatile when steam distilled. so it takes a long time Often used with thin-petaled flowers such as roses and jasmine.
- By placing flowers on a glass tray spread with a thin layer of animal fat. in order for the fat to absorb the aromatic substances from the flowers It takes about 1-3 days.
- This process is repeated until the fat absorbs enough aromatic compounds. The fat that absorbs this fragrance is called pommade. Then the pommade is dissolved in alcohol to get the essential oil out.
- The production of essential oils is usually more than 10% extracted in this way.

Solvent extraction

- This method yields highly concentrated essential oils. But the quality is not good because there are other substances mixed with other substances. Therefore, essential oils that have been used in the cosmetic and food industries are not popular.
- It is a method that can be used for plants that cannot tolerate high heat such as jasmine.
- The commonly used solvents are alcohol, petroleum ether. benzene or hexane Which will extract aromatic substances from plants, which will also contain wax, pigments and albumin The extracted substance is evaporated to expel the solvent at low temperature under a vacuum system to obtain the so-called concrete, which can be used to flavor soap. But it is not commonly used in perfumes because it is not pure enough
- The extracted essential oil is called absolute oil.




Cotton
Plant material
Cotton
Solvent
Solvent with essential oil

Expression/ Cold expression

- This method is often used with citrus plants such as oranges, lemons, kaffir limes, and pomelo.
- By squeezing the peel of the fruit, the plant cells are broken apart and the oil is released. Because this method of extraction does not use heat, the essential oils smell similar to that of fresh plants.
- The downside is that the resulting oil is small and impure.



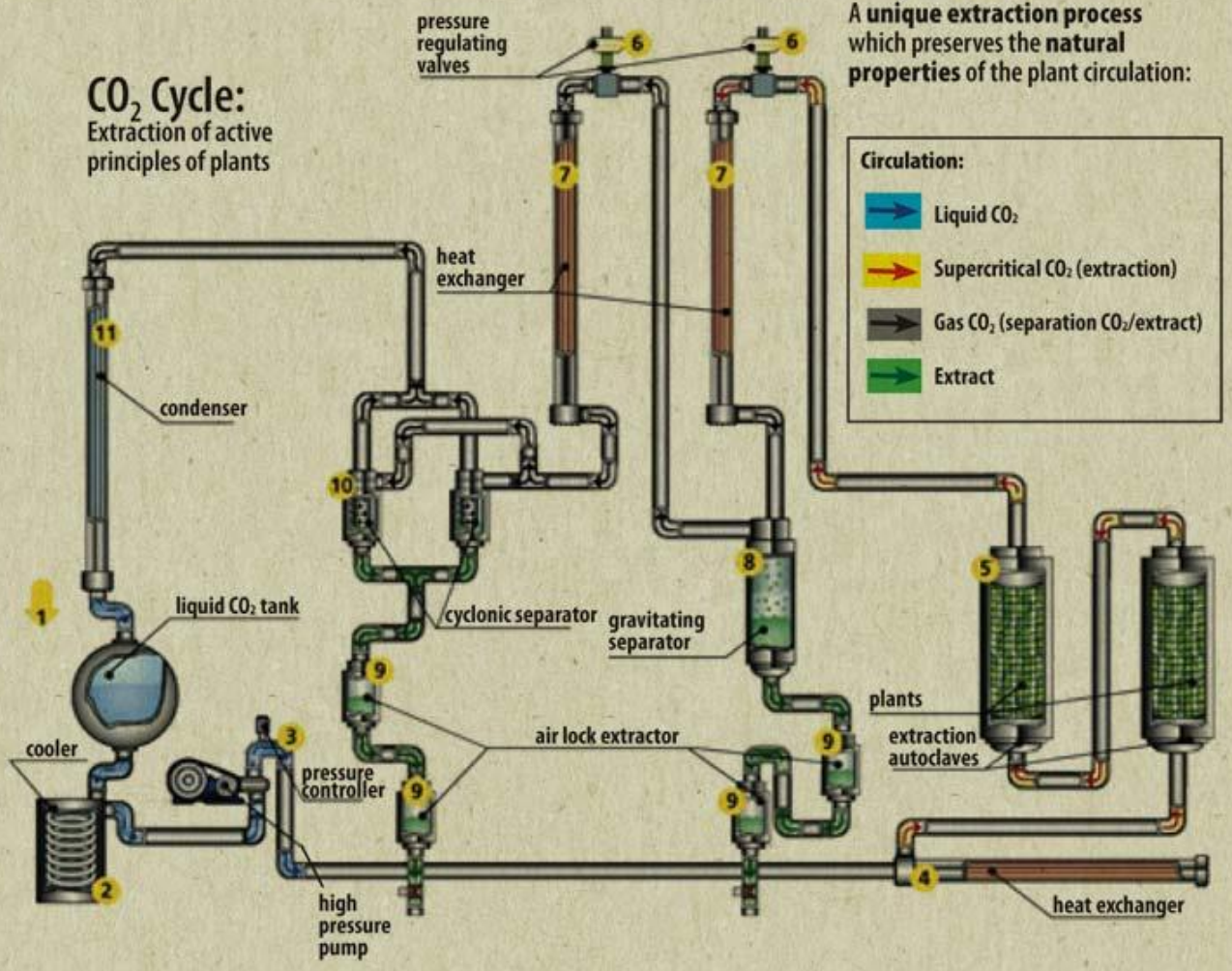
Fig. 8. Expression of the lemon oil from the rinds (Messina).

Super-critical carbon dioxide extraction

- This is a new way of extracting essential oils. By using liquid and gaseous carbon dioxide under high pressure and temperature. using a pressure of about 200 atm at a temperature of about 30 degrees Celsius
- The resulting essential oils are of good quality and high purity. But there is a disadvantage. The tools are very expensive.

THE SUPERCRITICAL EXTRACTION PROCESS

CO_2 Cycle:
Extraction of active principles of plants



CO_2 Extraction
A **unique extraction process** which preserves the **natural properties** of the plant circulation:

1. **CO_2 storage**
2. **Cooling:** CO_2 passes into a cold exchanger to maintain its liquid state before entering into the high pressure pump.
3. **Pressurization:** the pressure is raised to 300 bars.
4. **Reheating:** the temperature is raised to 31° C. CO_2 is supercritical.
5. **Extraction:** CO_2 supercritical is used as solvent to extract active plant ingredients without denaturing them.
6. **Relaxation:** Lowering of the pressure and thus return of the CO_2 to a gas state allows the separation of the extract from CO_2.
7. **Reheating:** The temperature is maintained at 30° C.
8. **Separation:** 1st separation stage: separation of CO_2 from the extract by gravity.
9. **Under pulling:** The extract is decompressed gradually to be under drawn in total safety.
10. **Cyclonic separation:** 2nd stage separation: Separation of CO_2 extract by centrifugal force.
11. **Liquefaction:** still in a gas state, CO_2 is cooled for liquefaction.

Essential Oil Quality Check

Several factors affect the quality of essential oils.

Plant type, species, origin

- Genetics mutation gene transfer
- Growth environment, temperature, humidity, exposure time, fertilizer, elevation, air, rainfall
- Plant growth stages, age, flowering stage, harvest season which affects the production of secondary substances of aromatic plants
- Plant parts used such as leaves, flowers, fruit bark, stem bark.
- How to store in fresh or dry condition
- Different extraction methods such as steam distillation, hydrodistillation, expression
- Containers used for storage such as brown glass bottles, aluminum bottles, plastic bottles, cork.

Essential Oil Quality Check

It is an inspection of various external features. Both physical, chemical, physical, including Adultation

Quality testing of essential oils By examining both chemical and physical properties as follows

1. Examination of general characteristics such as color, clarity - turbidity, odor, suspended sediment, etc.
2. Examination of physical properties such as solubility in ethanol Determination of relative density. Determination of optical rotation. of essential oils Determination of the refractive index of essential oils
3. Chemical characterization using gas chromatography method

Inspecting general characteristics

•**Color**	It meets the requirements of each type. It may or may not be colored.
•**clarity - turbidity**	Must be clear, no turbid precipitation
•**Smell**	Can be tested by smelling and has a specific smell
•**Suspended sediment**	no suspensions this may mean that the extract is poorly extracted or contains contaminants

Physical characterization

- **solubility in ethanol to see what concentration of essential oils can be dissolved in ethanol. How much is it? Test Method Comply with ISO 875-1981 Essential oils - Evaluation of miscibility in ethanol on test methods for dissolution in ethanol.**

- **Determination of relative density t**o determine the density of essential oils compared to the density of water at 20°C, in accordance with ISO 279-1981 Essential oils - Determination of relative density at 20OC (Reference Method) on the relative density test method at 20°C.

- **Determination of optical rotation of essential oils b**y measuring the angle of deviation of polarized light that shines through essential oils The test method shall comply with ISO 592-1981 - Determination of optical rotation on test methods for optical rotation at 25 °C.

- **Determination of the refractive index of essential oils** by measuring the angle of refracted light When exposed to light, essential oils were tested according to ISO 280-1976 - Determination of refractive index on the refractive index test method at 20 °C.

Chemical characterization

acid value analyzed by chemical reaction

ester value analyzed by chemical reaction

carbonyl value analyzed by chemical reaction

ketone value analyzed by chemical reaction

The amount of 1,8-cineole was analyzed by chemical reaction.

The menthol content (in peppermint oil) was determined by chemical reaction according to USP requirements.

The content of dimethyl sulfide (in peppermint oil) was determined by chemical reaction according to USP specifications.

The heavy metal content (in peppermint oil) was determined by chemical reaction according to USP requirements.

Analysis by Thin Layer Chromatography (TLC)

Analysis by gas chromatography (Gas Chromatography, GC)

Chemical characterization

- It is an analysis of various components. in each essential oil using gas chromatography method
- By injecting a small amount of essential oil (~0.1 ml) into a gas chromatography (GC) apparatus consisting of the following key components: Injector, Oven, Column, Detector, Recorder, Integrator
- The result is shown in the form of Chromatogram

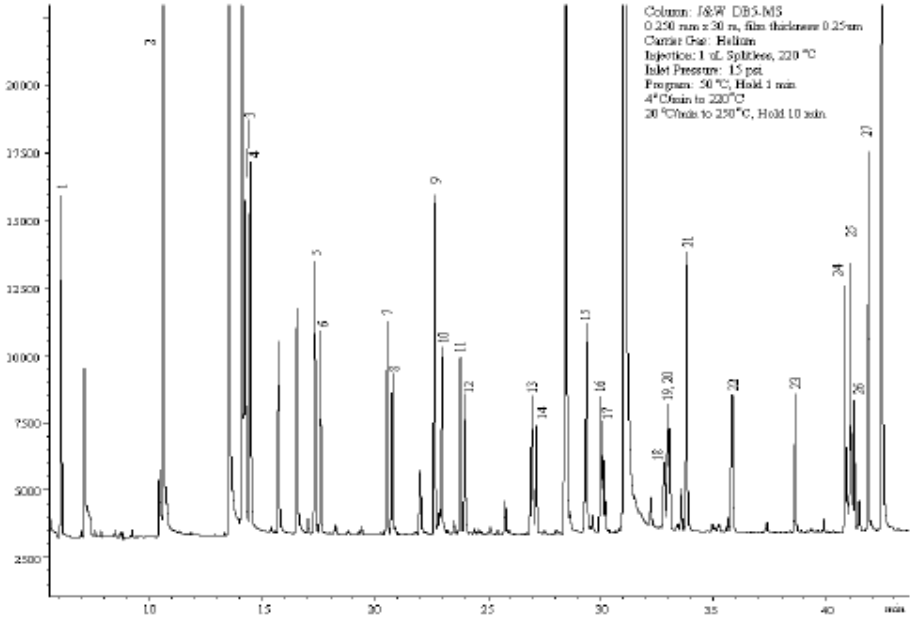

Column: J&W DB5-MS
0.250 mm x 30 m, film thickness 0.25um
Carrier Gas: Helium
Injection: 1 uL Splitless, 220 °C
Inlet Pressure: 15 psi
Program: 50 °C, Hold 1 min
4°C/min to 220°C
20 °C/min to 250°C, Hold 10 min
20000
17500
15000
12500
10000
7500
5000
2500
10
15
20
25
30
35
40
min

Figure 1

An agency that sets standards for essential oils.

1. The International Organization for Standardization (ISO)
2. The Essential Oil Association of the United States (EOA)
3. he International Fragrance Association (IFRA), with The Research Institute for Fragrance Materials (RIFM), is responsible for biological information such as toxicity, carcinogenicity and pharmacological activity.
4. The Food and Drug Administration of the United States (FDA) and The Favor and Extracts Manufacturer's Association (FEMA) study the safety of essential oils in products.
5. Thai Industrial Standards Institute (TISI), Ministry of Industry

ISO Standard Requirements for Essential Oil Quality Control

- Relative density 20 °C : ISO 279-1981, ISO 279-1998
- Refractive index 20 °C : ISO 280-1976 , ISO 280-1998
- Optical rotation 20 °C : ISO 592-1981 , ISO 592-1998
- Miscibility in ethanol : ISO 875-1981 , ISO 875-1999
- Ester value : ISO 1241-1980 , ISO 709-2001
- Acid value : ISO 1242-1999
- Carbonyl compounds content : ISO 1279-1984

Thai standards

Faculty Academic Committee 861 Essential Oil Industry Standards has set 7 standards as follows:

1. Industrial Product Standard Phlai Oil Standard No. TIS 1679-2541
2. Industrial product standards Clove Bud Oil Standard No. TIS 1680-2541
3. Industrial Product Standard Lemongrass Oil Standard No. TIS 1681-2541
4. Industrial product standards Citronella Oil Standard No. TIS 1682-2541
5. Industrial product standards Kaffir lime skin oil (Makrut Peel Oil) Standard No. TIS 2078-2544
6. Industrial product standards Kaffir lime leaf oil (Makrut Leaf Oil) Standard No. TIS 2079-2544
7. Industrial Product Standard Basil Oil (Thai type) Standard No. TIS 2080-2544

Scope of the standard

- Definition, botanical name
- The Characteristics :
  - General characteristics that must be clear liquid. Specifies the display color. Free from sediment and suspended solids No separation of oil layers with a unique smell
  - Physics feature such as solubility in ethanol relative density optical rotation, refractive index, etc.
  - Important chemical constituents in order to make essential oil products from various herbs Good quality suitable for use

Packing designation which controls the container to be dry, clean, sealed and not reacting to essential oils if using glass containers must be protected from light And there must be 5-10 percent of the container's free space left in the container. The net weight must not be less than that indicated on the label. as well as the display of marks and labels must be easily seen legible More importantly, details must be provided as required by the standard.

Conclusion

- The raw materials used in the extraction of essential oils are from different parts of the plant, namely leaves, roots, flowers, cores, fruits, and seeds, each of which yields different amounts of oil. can be grouped little oil group medium oil group and a lot of oil
- There are five methods of extracting essential oils from raw materials, including steam distillation. Chemical use, fermentation, liquid carbon dioxide extraction and oppression Each method is suitable for the type of material to be extracted. And each method has a different effect on the purity of essential oils. It is necessary to select an extraction method suitable for the type of plant, the part used, and the available budget.
- Monitoring the quality of essential oils is essential. because it is used to confirm the quality of essential oils Users need to study in order to select essential oils that are suitable for use.

Assignment

- Ask students to search for videos about essential oil extraction and select one clip.
- Submit an edited original clip together with a summary of the content learned from the video clip and the reason for selecting this clip.
- Recording an individual presentation clip and sending it to google classroom