



SugandhimTM

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Technology of **Essential Oils**

VOLATILE OILS

Volatile oils are products which are generally complex in composition, consisting of the volatile principles contained in plants, and are more or less modified during the preparation process.

Only 2 procedures may be used to prepare official oils

- i. Steam distillation**
- ii. Expression**

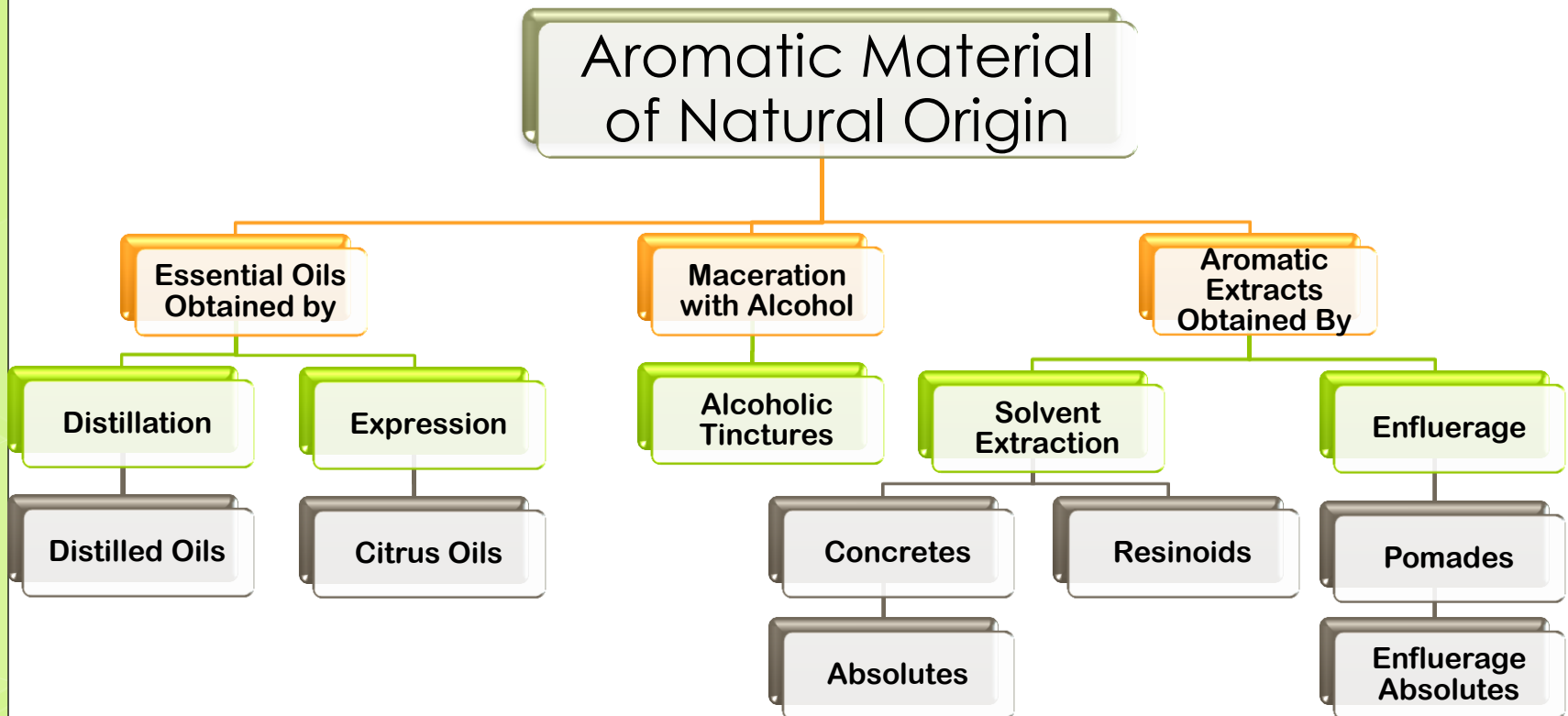
4 Main types of volatile oils

- i. Concretes**
- ii. Pomades**
- iii. Resinoids**
- iv. Absolutes**
- v. Essential Oils**

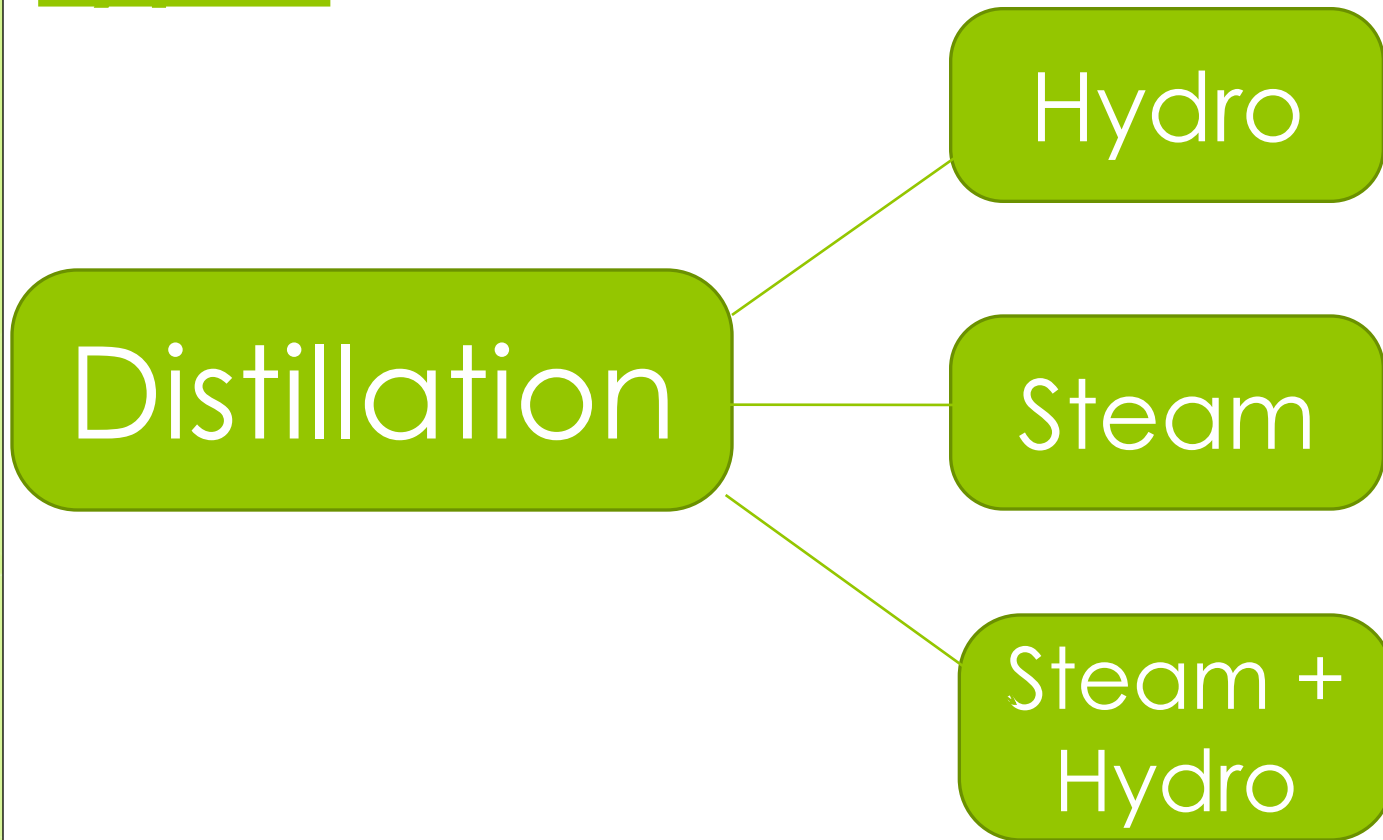
Extraction Techniques

- Distillation Techniques
- Special Distillation Technique
- CO₂ Extraction
- GCMS Comparison of CO₂ Extracted Oil

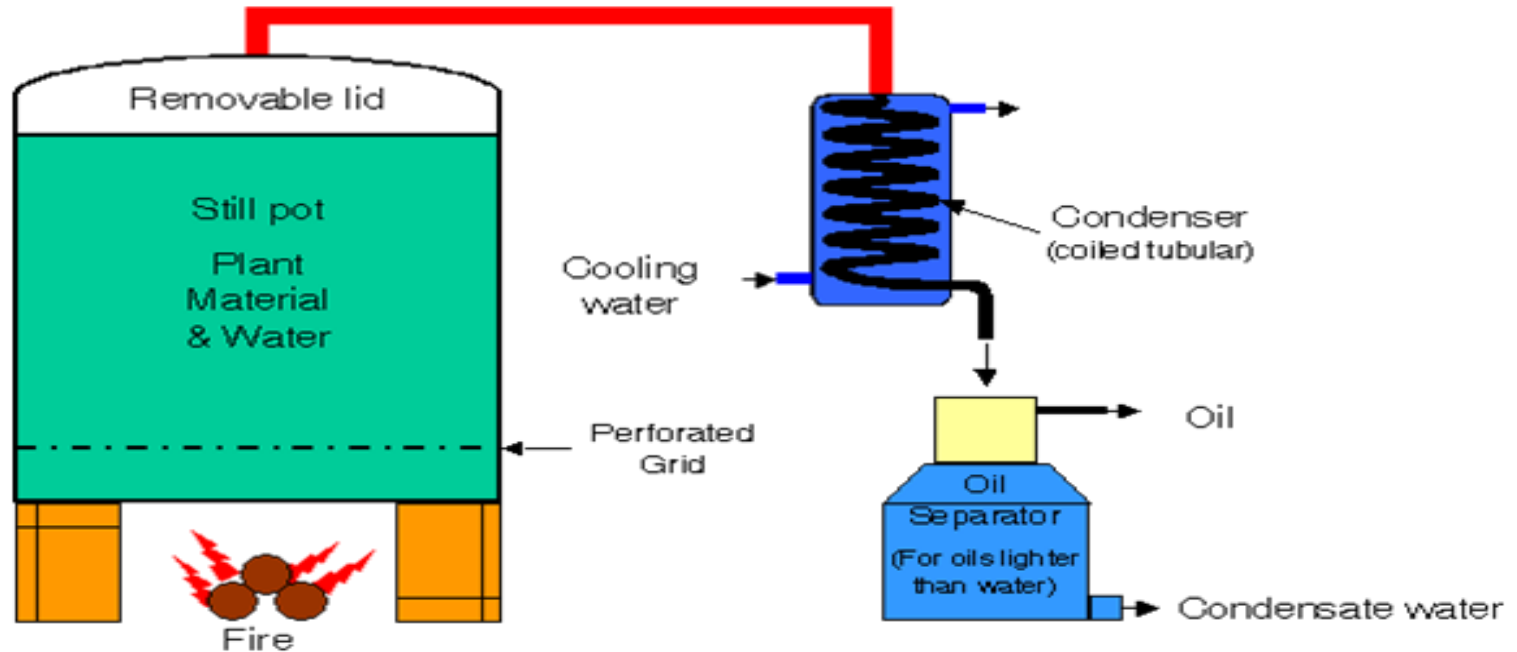
Methods of Oil Extraction



Types



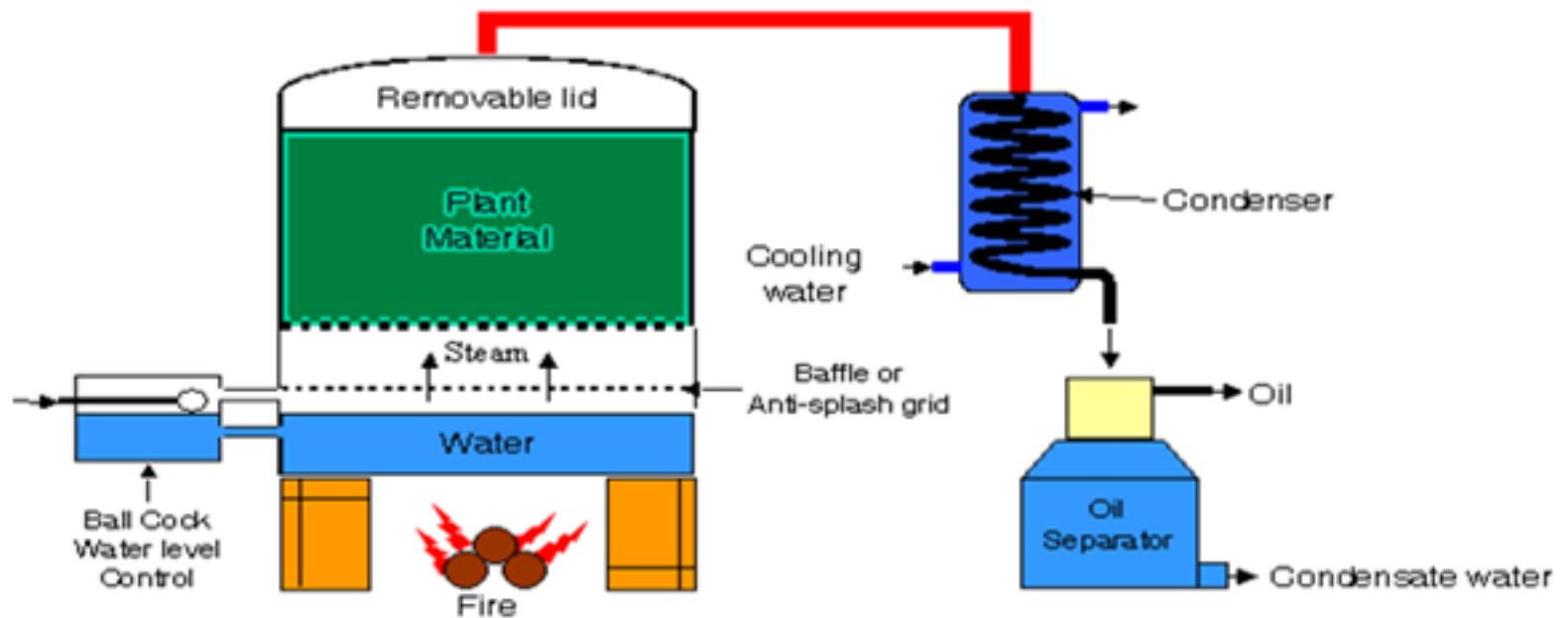
Hydrodistillation



- Herbs Are Put In Water & Distillation Vessel & Is Heated From Bottom.
- Most Flowers Are Distilled By This Methods .
- Eg :- Rose , Ylang Ylang. Tagate

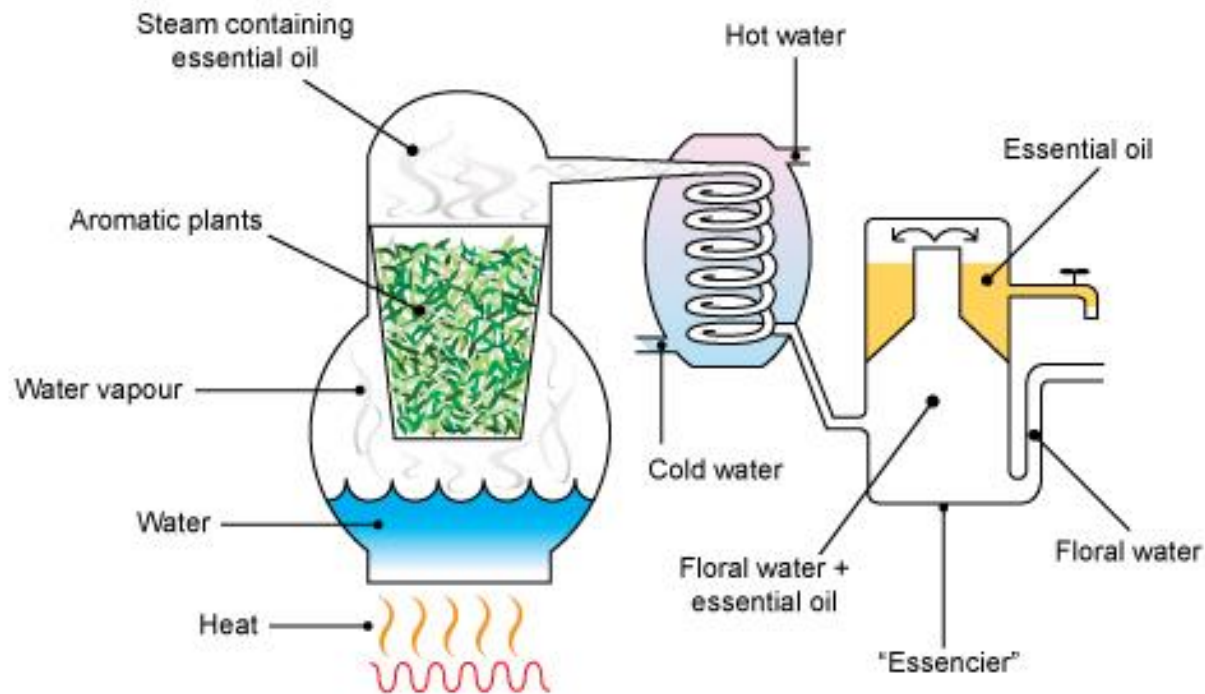
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- Water : its quantity in the still must always be sufficiently more ,otherwise the plant material can over-heat and char.
 - Plant material : it must be kept agitated as the water boils otherwise it may settle in the bottom of the still and become damaged by the heating.
 - Chopping or grinding the material into fine particles may help to keep the material dispersed in the water.
 - It is very easy for still 'off-notes' to be generated, since some components of the oil are more susceptible to chemical change and oxygenated components tend to dissolve in the still water.
 - Some plant materials like cinnamon bark contain high levels of mucilages and as these are leached out , the viscosity of the water increases and there is a high risk of charring.
 - The stills tend to be small and therefore it will take a long time to accumulate much oil and each batch may be highly variable containing better quality oil mixed with poor quality.
 - Water distillation is a slower extraction process than the other two distillation types and therefore less energy efficient.

Hydro-Steam Distillation



- In this live steam is injected in herbs kept in water.
- Leaves are normally distilled by this method.
- Eg :- Lemongrass , Palmrosa, Geranium.

Steam Distillation



- Roots & Woods After Crushing Are Used For Steam Distillation.
- Eg :- Sandalwood , Vetiver , Nagarmotha
- Yield Of Oil Is More Than Other Two Type Of Distillation.

Steam distillation

ADVANTAGES OF STEAM & HYDRO DISTILLATION OVER HYDRO DISTILLATION

- Higher oil yield.
- Possible to achieve a high packing density because the plant material is not suspended in the water.
- Oil component less susceptible to change due to wetness and thermal conductivity of the still from the heat source.
- The effect of refluxing is minimised.
- Oil quality more reproducible.
- Faster process so more energy efficient.

ADVANTAGES & DISADVANTAGES OF STEAM DISTILLATION

- The amount of steam and the quality of the steam can be controlled.
- Lower risk of thermal degradation as temperature generally not above 100 °C.
- Most widely used process for the extraction of essential oils on a large scale.
- Throughout the flavour and fragrance supply industry it is the standard method of extraction.
- Requires higher level of technical skill and fabrication and repairs and maintenance require a higher level of skill.

Parameters Affecting the Oil Yield

- ◉ Mode Of Distillation
- ◉ Materials of Fabrication of Equipment
- ◉ Condition of Raw Material
- ◉ Time for Distillation
- ◉ Loading of Raw Material and Steam Distillation
- ◉ Operating Parameters
- ◉ Condition of Tank and Equipment

Site

- ◉ Availability of adequate water .
- ◉ Energy source: electricity, boiler fuel.
- ◉ Easy transport access
- ◉ Skilled and unskilled labour
- ◉ Close proximity to plant material
- ◉ Access to fabricators and machine shop for repairs
- ◉ Environmental zoning, plant waste and waste water discharge

Distillation Still



Stainless Steel



Mild Steel



Copper is material used from ancient times till today .

There are few following

advantage in same

A) it can be repaired on site

B) with time copper appreciate

C) good conductor of heat

D) quality of oil distilled in same is found to be better & have sweetness in same so liked in Aromatherapy.

Glass is another material of construction but due to it's brittleness same is common in lab scale not on commercial scale.

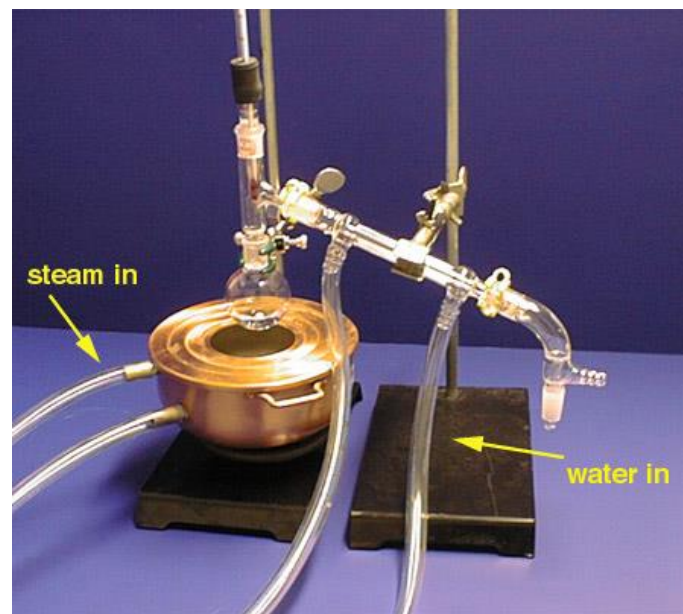
Boiler

- Should produce enough steam to adequately remove the oil from the plant material
- Temperature is very important for oil's quality as well yield.
- Low temperature leads to undesired reaction like saponification , condensation takes place.
- Also color of oil is lighter if temperature is low.



Condenser

- The role of the condenser is to change the oil and water vapour back to a liquid
- Two main types: coiled tube or multitube
- cooling system where water is used as coolant is very important.
- If temperature goes up un condensed vapour escapes without getting condensed.
- This results in loss of yield + partial oil.



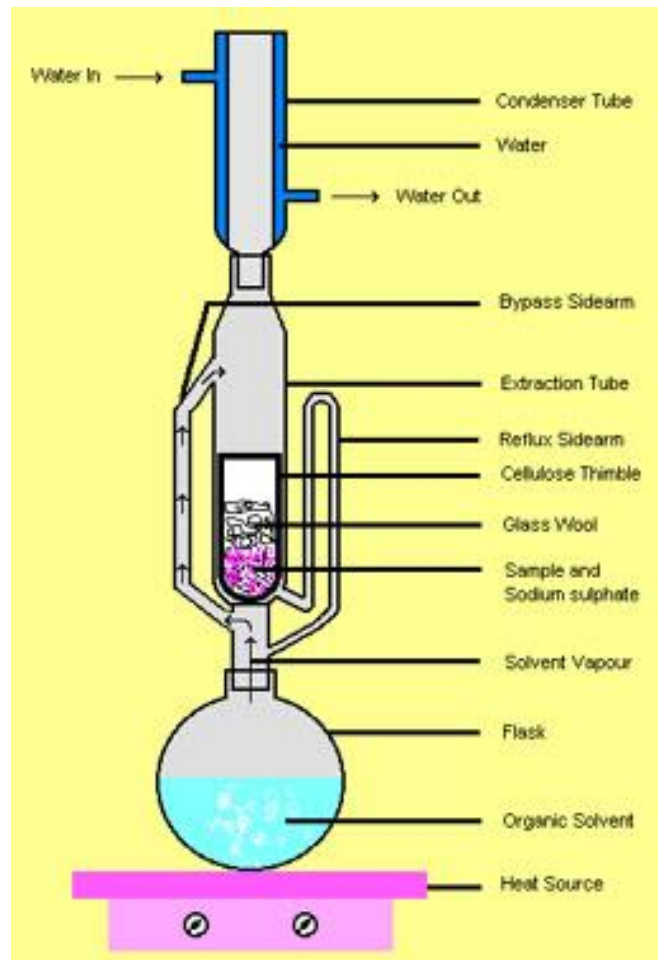
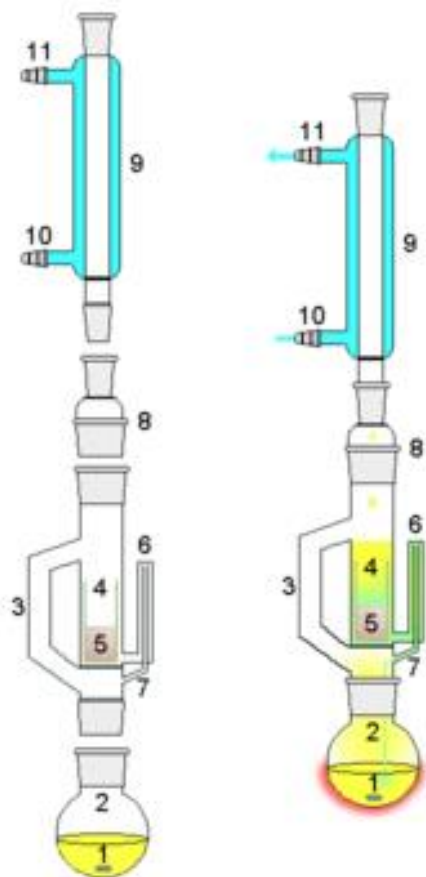
Oil Separator

- Design of separator depends on density of the oil (if <1 , oils are lighter than water and float; if >1 , oils heavier than water and sink). Only a few wood and root oils are heavier than water
- Large enough capacity to allow the oil particles to form droplets and readily separate from the water (recommended at least a 4 minute retention time in the separator before out flow)
- Controlling temperature can be used to improve separation
- Seek professional advice on design as poor separation affects all the effort of distillation to extract oil

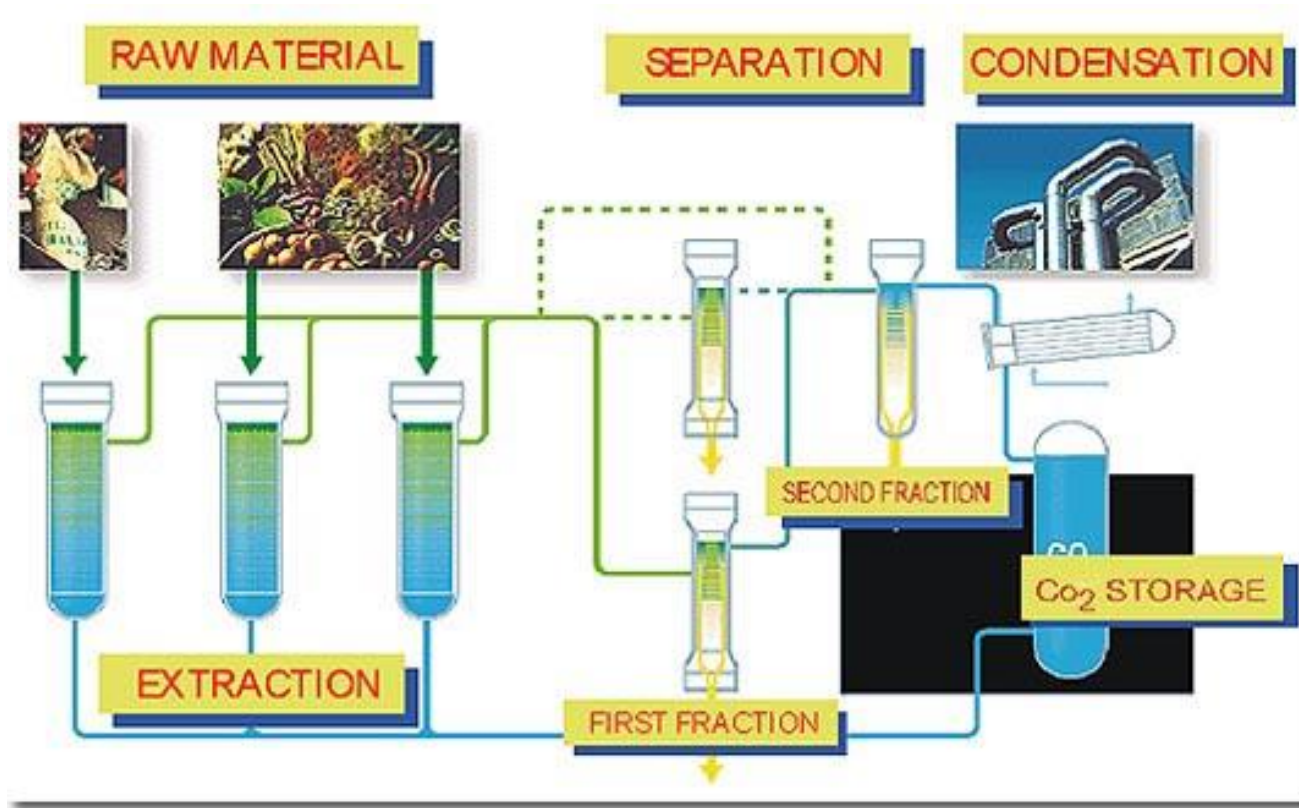
Storage of Oil

- System to filter separated oil
- Storage in suitable containers that exclude light
- Method to remove dissolved water (filtered bed of anhydrous sodium sulfate or chilling)
- Removal of residual still notes and dissolved oxygen (bubbling stream of nitrogen or allow oil to breathe and topping drum to over flowing to remove all air)

SOXHLET EXTRACTION – Coriander Oil

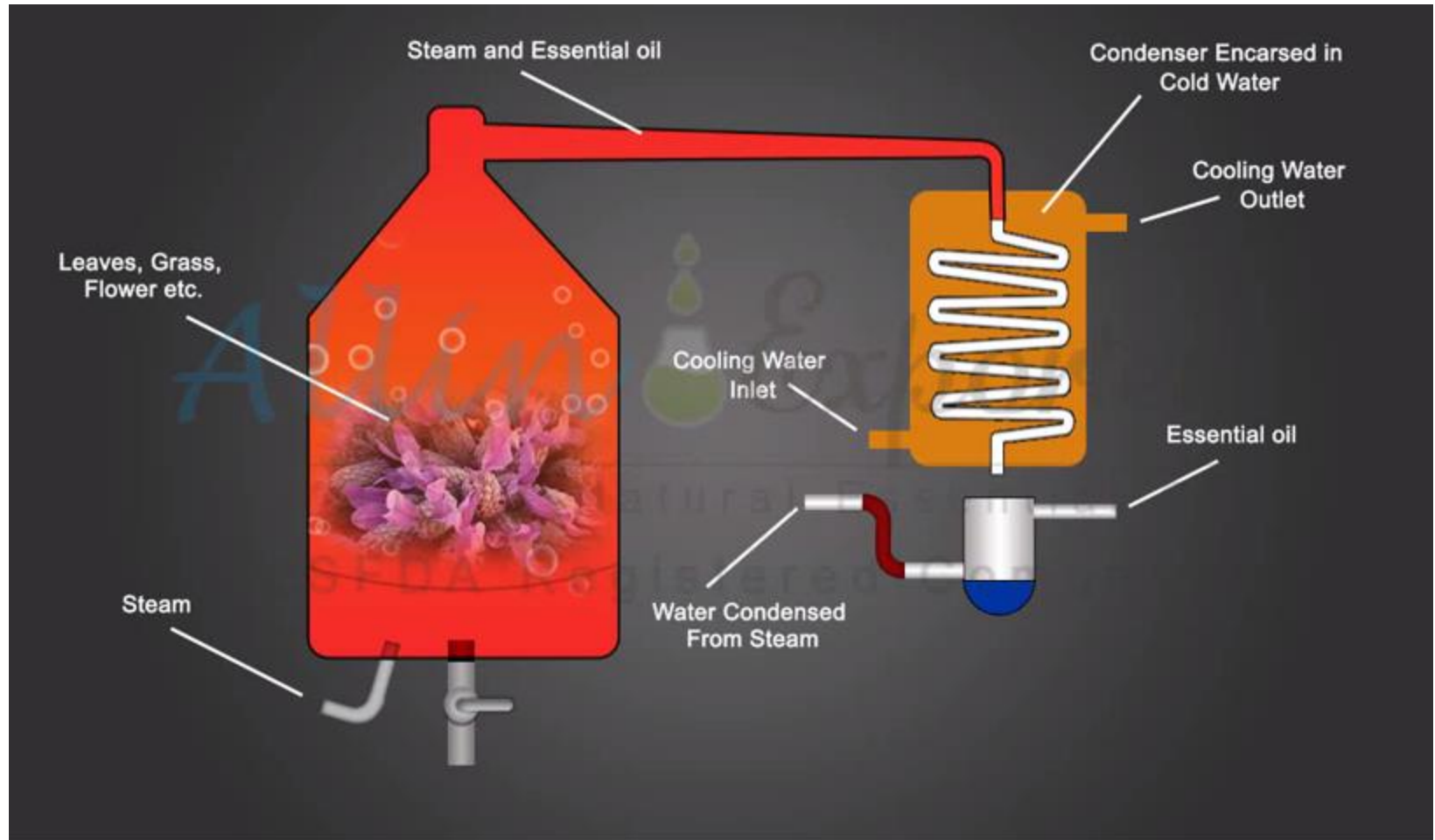


CARBON DIOXIDE / CO₂ EXTRACTION



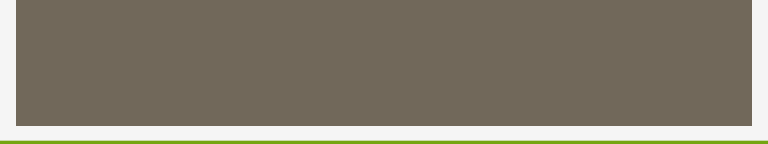
Solvents of supercritical fluid extraction

- The choice of the SFE solvent is similar to the regular extraction. Main considerations .
- Good solving property .
- Inert to the product.
- Easy separation from the product .
- Economical .



Fluid	Critical Temperature (K)	Critical Pressure (bar)
Carbon dioxide	304.1	73.8
Ethane	305.4	48.8
Ethylene	282.4	50.4
Propane	369.8	42.5
Propylene	364.9	46.0
Trifluoromethane (Fluoroform)	299.3	48.6
Chlorotrifluoromethane	302.0	38.7
Trichlorofluoromethane	471.2	44.1
Ammonia	405.5	113.5
Water	647.3	221.2
Cyclohexane	553.5	40.7
n-Pentane	469.7	33.7
Toluene	591.8	41.0

Critical Conditions for Various Supercritical Solvents

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- Use of supercritical carbon dioxide extraction .
 - Expensive yields good quality oils .
 - Carbon dioxide becomes supercritical at 31 degrees Celsius, which is a state in which it is not really gas or liquid, but has qualities of both.
 - Excellent solvent for extraction of essential oils.
 - Low temperature required & process is instantaneous.
 - The carbon dioxide is furthermore inert and therefore does not chemically interact with the essence.
 - To remove the carbon dioxide solvent, you simply need to remove the pressure under which it is kept.
 - This process has to take place in a closed chamber for the supercritical pressure required for carbon dioxide is 200 atmospheres - that is 200 times the pressure of normal atmosphere.
 - To achieve this type of pressure heavy-duty stainless steel equipment is required.
 - High capital investment.

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- **CO₂ is the most widely used fluid in SFE. However, water is the other increasingly applied solvent. One of the unique properties of water is that, above its critical point (374°C, 218 atm.), it becomes an excellent solvent for organic compounds and a very poor solvent for inorganic salts.**
 - **This property gives the chance for using the same solvent to extract the inorganic and the organic component respectively.**

Cardamom Oil Different Between CO₂ & Steam Distilled By GCMS

PK	Compound name	CO2	NON CO2
1	Eucalyptol	2.4343	28.011
2	Alpha-Terpinyl acetate	59.4741	26.8498
3	Limonene		9.476
4	Terpineol <alpha->	3.0678	5.9816
5	Linalyl acetate	2.927	5.8141
6	Pinene <alpha->		2.8154
7	Linalool	0.8267	2.4348
8	Terpinen-4-ol	0.7818	1.9829
9	Sabinene	0.0684	1.5823
10	Pinene <beta->		1.4099
11	Nerolidol <(E)->	5.7522	1.3355
12	Myrcene		1.2255
13	Geranyl acetate	2.3096	1.2065
14	Cymene <para->		0.8287
15	Butyraldehyde <3-(5-methyl-, 2-furyl)->	1.885	0.7816
16	Bornyl acetate	0.0769	0.7772
17	Isobornyl isobutanoate <6-hydroxy->	3.1621	0.6693
18	Terpinene <gamma->	0.3628	0.4805
19	Terpinene <alpha->		0.4757
20	Fernesene <(E)-, beta->	1.1908	0.3472
21	Caryophyllene <9-epi-(E)->	1.5764	0.291
22	Isobornyl isobutanoate <5-hydroxy->	0.5014	0.2255
23	Selinene <beta->	1.6105	0.2058
24	Sabinol <trans->	0.1354	0.0214
25	Sesquicineole <dehydro->	0.2402	0.0101
26	Linalyl phenylacetate	0.7492	
27	Eicosene <1->	0.7164	
28	Cadinene <gamma->	0.7124	
29	Octadecane <n->	0.3698	
30	Hexadecanoic acid <n->	0.365	
31	Heptadecanol <n->	0.2024	
32	Hexadecane <n->	0.1974	
33	Eicosane <n->	0.1332	
34	Tricos-(9Z)-ene	0.1269	

Analysis of Ginger Oil CO₂ By GCMS

PK	Compound name	Area Pct
1	Zingiberene <alpha->	36.514
2	Bisabolene < Z > gamma	13.760
3	Sesquiphellandrene <beta->	12.684
4	Curcumene <alpha->	7.703
5	Sabinene	4.95
6	Camphene	3.599
7	Zingerone	1.899
8	Germacrene D	1.620
9	Pinene <alpha->	1.177
10	Farnesene <(E)-, beta->	1.148
11	Borneol	1.146
12	Eugenol acetate <dihydro->	1.130
13	Germacrene B	0.961
14	Germacrene A	0.883
15	Myrcene	0.79
17	Terpinolene	0.244
18	Ethyl chrysanthemumate <trans->	0.228
19	Premnaspirodien	0.223
20	Undecan-2-one	0.216
22	Decanal <n->	0.128
23	Gurjunene <alpha->	0.125
24	Nona-1,3,7-triene <4,8-dimethyl-, (E)->	0.116
25	Camphor	0.115
26	Camphene hydrate	0.095
27	Isobornyl acetate	0.089
28	Tricyclene	0.07

Analysis of Clove Bud Oil CO₂ By GCMS

PK	Compound Name	Area Pct
1	Eugenol	75.7806
2	Caryophyllene <(E)->	10.2018
3	Isoeugenol	8.14
4	Humulene <alpha->	2.0913
5	Cadinene <delta->	0.5098
6	Baeckeol	0.4628
7	Cinnamaldehyde <4-hydroxy-3-methoxy->	0.4113
8	Caryophyllene oxide	0.3255
9	Selina-3,11-dien-6-alpha-ol	0.2219
10	Coniferyl alcohol <E->	0.1725
11	Cadinene <gamma->	0.1494
12	Estragole	0.1367
13	Cadalene	0.1171
14	Caryophyllene alcohol	0.1073
15	Salicylate <methyl->	0.1072
16	Isoeugenyl phenylacetate	0.0872
17	Selinene <alpha->	0.0618
18	Amorpha-4,7(11)-diene	0.0549
19	Eucalyptol	0.0506
20	Humulene epoxide II	0.05
21	Anethole <(Z)->	0.0494
22	Selinene <beta->	0.0441
23	Tetracosane <n->	0.0331

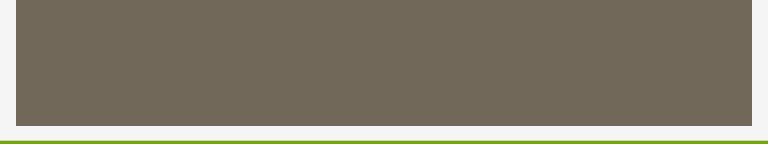
Compare distilled oils and solvent extracted oils (ex: rose)

A) Hydro distillation:

Rose being very delicate hydro distillation with controlled heat is recommended in order to prevent loss of high volatile component which are responsible for nice odour.

B) Solvent extraction:

- A Common method for capturing & recovering volatile component high boiling sesquiterpenes.
- Resultant product from solvent extraction of Rose flower is concrete which is processed further to remove wax & obtain absolutes.
- Smell of both products rose absolute & hydro distilled oil is very different



THANK YOU!