




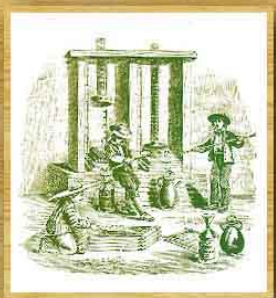
## Hydro-distillation and steam distillation from aromatic plants

Sudeep Tandon  
Scientist  
Chemical Engineering Division  
CIMAP, Lucknow




## HISTORY

- Written records of herbal distillation are found as early as the first century A.D., and around 1000 A.D., the noted Arab physician and naturalist Ibn Sina also known as Avicenna described the distillation of rose oil from rose petals
- The ancient Arabian people began to study the chemical properties of essential oils & developed and refined the distillation process
- Europeans began producing essential oils in the 12th century

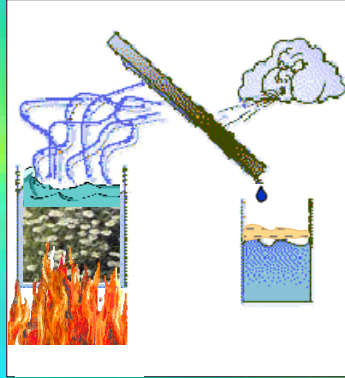



A traditional method of extracting oil involved subjecting the plants to pressure



## DISTILLATION ?

- A process in which a liquid or vapour mixture of two or more substances is separated into its component fractions of desired purity, by the application and removal of heat.
- In simple terms distillation of aromatic herbs implies vaporizing or liberating the oils from the trichomes / plant cell membranes of the herb in presence of high temperature and moisture and then cooling the vapour mixture to separate out the oil from water.
- It is the most popular widely used and cost effective method in use today for producing majority of the essential oils throughout the world
- *Distillation is an art and not just a "Chemical" process that is reliant upon many factors for successful quality oil production.*



## BASIC SCIENTIFIC PRINCIPLES INVOLVED IN THE PROCESS

- ▶ To convert any liquid into a vapour we have to apply energy in form of heat called as latent heat of vaporization
- ▶ A liquid always boils at the temperature at which its vapour pressure equals the atmospheric / surrounding pressure
- ▶ For two immiscible liquids the total vapour pressure of the mixture is always equal to the sum of their partial pressures
- ▶ The composition of the mixture will be determined by the concentration of the individual components into its partial pressure
- ▶ As known the boiling point of most essential oil components exceeds that of water and generally lies between 150 – 300°C

- ▶ If a sample of an essential oil having a component 'A' having boiling point for example 190°C and the boiling point of the water is 100°C. when the two immiscible liquids are brought together, and once their vapor have reached saturation, the temperature will immediately drop to 99.5°C, which is the temperature at which the sum of the two vapor pressure equals 760 mm Hg.

$$P_{\text{TOTAL}} = P_{\text{A}} + P_{\text{WATER}}$$

- ▶ Therefore In a mixture of water and EO saturated vapour above the mixture is :

$$\frac{\text{Wt of 'A'}}{\text{Wt. of water}} = \frac{\text{Vapor pressure of 'A' X mol. Wt. of the 'A'}}{\text{Vapor pressure of water X Mol. Wt. of water}}$$

- ▶ Thus any essential oil having high boiling point range can be evaporated with steam in a ratio such that their combined vapour pressures will be equal to the atmospheric pressure and can be isolated from the plants by the wet distillation process

## EXTRACTION OF ESSENTIAL OILS BY DISTILLATION IS GOVERNED BY

- **SENSITIVITY OF THE ESSENTIAL OIL TO THE ACTION OF HEAT AND WATER.**

*Essential oils with high solubility in water and which are susceptible to damage by action of heat cannot be steam distilled.*

- **VOLATILITY OF THE ESSENTIAL OIL**

*The oil must be steam volatile for steam distillation to be feasible.*

- **WATER SOLUBILITY OF THE ESSENTIAL OIL**

*Most of the essential oils of commerce are steam volatile, reasonably stable to action of heat and practically insoluble in water and hence suitable for processing by steam distillation*

## TECHNIQUES OF DISTILLATION

- ❖ Water/hydro distillation
- ❖ Steam and water distillation
- ❖ Direct steam distillation
- ❖ Distillation with cohobation
- ❖ ...Hydro Diffusion



## ESSENTIAL OIL ESTIMATION : THE CLEVANGER APPARATUS

- ❖ USED FOR ESTIMATION OF ESSENTIAL OIL CONTENT IN AROMATIC PLANTS ON BENCH SCALE
- ❖ ALSO USEFUL FOR PRELIMINARY QUALITY EVALUATION OF OIL QUALITY

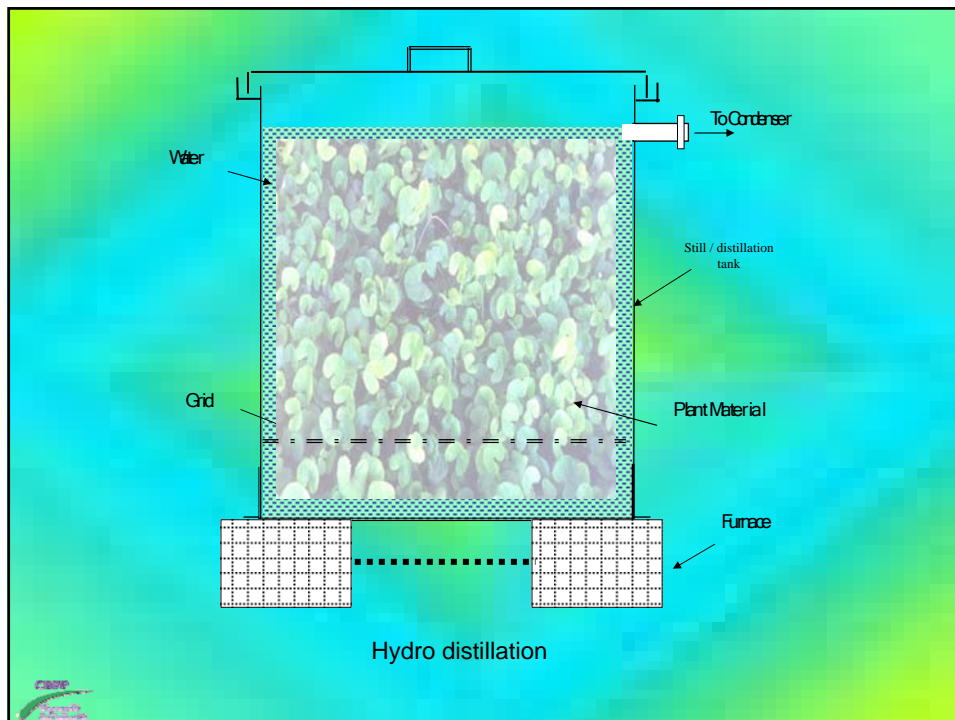


## HYDRO DIFFUSION

- Unlike traditional steam distillation, hydro diffusion works off the diffusion principle of allowing steam to enter the top of the plant charge and diffuse its through the charge by gravity.
- The process uses principle of osmotic pressure to diffuse oil from the oil glands.
- The system is connected and low pressure steam is passed into the plant material from a boiler from the top.
- The condenser, generally of tubular construction which is directly under the basket within the still, is used for cooling
- The oil and water are collected below the condenser in a typical oil separator.
- It would appear that hydro diffusion is an efficient process to use as it gives good yield of the oil
- But due to co extraction of other non volatiles and polar components with the essential oils complicated the process

## WATER / HYDRO DISTILLATION

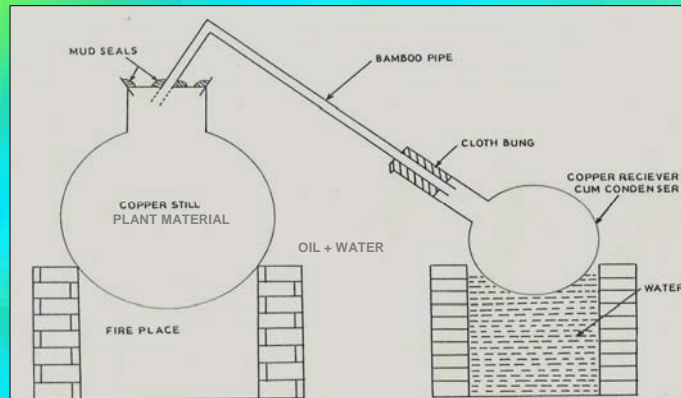
- ✘ Hydro / water distillation is one of the simplest, oldest and primitive process known to man for obtaining essential oils from plants.
- ✘ Mostly used by small scale producers of essential oils
- ✘ In water / hydro distillation the plant material is almost entirely covered with water as suspension in the still which is placed on a furnace
- ✘ Water is made to boil and essential oil is carried over to the condenser along with the steam.
- ✘ Useful for distillation of powders of spices and comminuted herbs etc
- ✘ The Deg Bhabka method of India using copper stills is an example of this technique



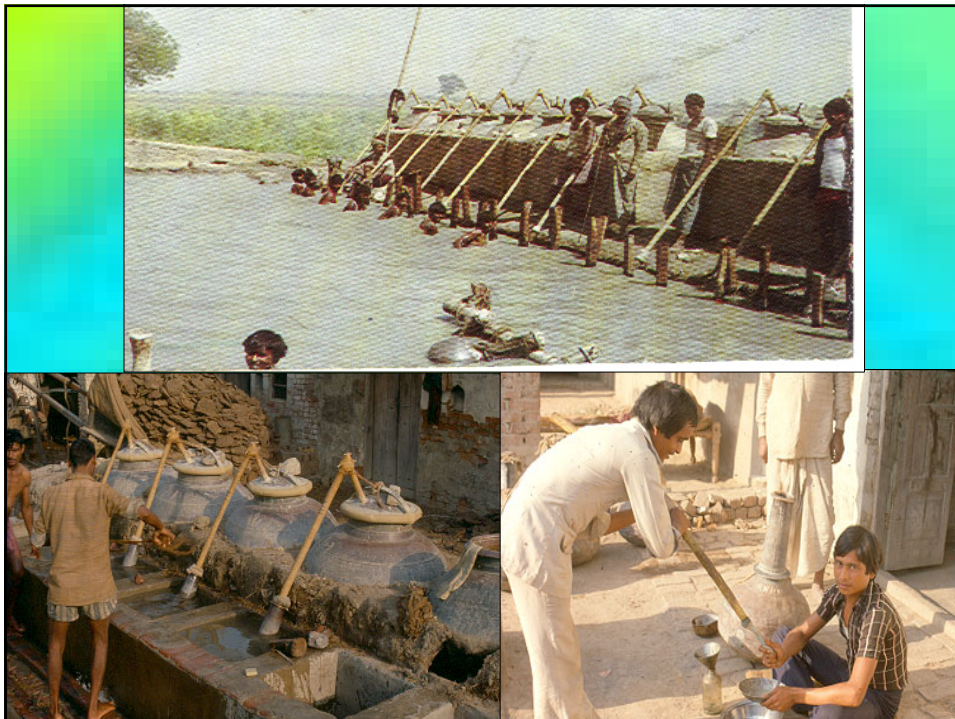
## DISADVANTAGES OF THE HYDRO DISTILLATION

- ➔ The process is slow and the distillation time is much longer thereby consuming more firewood / fuel making process un economical.
- ➔ Variable rate of distillation due to difficult control of heat
- ➔ Extraction of the herb is not always complete
- ➔ As the plant material near the bottom walls of the still comes in direct contact with the fire from furnace there is a likelihood of its getting charred and thus imparting an objectionable odor to the essential oil
- ➔ Prolong action of hot water can cause hydrolysis of some constituents of the essential oils such as esters etc which reacts with the water at high temperatures to form acids & alcohols
- ➔ Not suitable for large capacity / commercial scale distillations
- ➔ Not suitable for high boiling hardy roots / woody plant materials

## TRADITIONAL DEG / BHABKA METHOD



- ✳ **OLDEST AND MOST PRIMITIVE METHOD**
- ✳ **STILL BEING USED FOR MANUFACTURING OF ROOH, ATTARS IN INDIA**
- ✳ **ROOH IS THE PURE ESSENTIAL OIL WHEREAS ATTAR IS THE ESSENCE OF THE HERB TRANSFERRED INTO A FIXATIVE OIL MAINLY SANDALWOOD OIL**
- ✳ **BEING USED IN AREAS OF KANNUAJ FOR MAKING ROSE, JASMINE, KEWDA KHUS, KESAR ATTARS.**



## STEAM AND WATER DISTILLATION

- ⇒ **To overcome the drawbacks of the water distillation, modifications in techniques was developed**
- ⇒ **Also sometimes referred as wet steam distillation**
- ⇒ **The plant material is supported on a cage / perforated grid below which water is boiled**
- ⇒ **Direct contact of plant material with hot furnace bottom is thus avoided.**
- ⇒ **The water below the grid is heated by open fire which produces saturated and wet steam which rises through the plant material vaporizing the essential oil with it**
- ⇒ **Advantages : High oil & reproducible yields, faster, lesser fuel**

## STEAM AND WATER DISTILLATION.....

- ▶ **The field distillation / portable / directly fired type units based on this techniques**
- ▶ **Due to their very simple construction, low cost and easy operation field distillation units are extremely popular with essential oil producers in developing countries**
- ▶ **Such field units generally have capacities to hold 100 kg to 2000 kg plant material.**
- ▶ **Furnace is mostly fueled by locally available fire wood, straw or spent and dried plant material which is being distilled.**



**STEAM AND WATER DISTILLATION**

*Directly fired type : field distillation units*

**DISADVANTAGES**

- *Unsafe*
- *Time consuming as low pressure steam*
- *Poor quality oil*
- *Improper condensation*
- *Oil separation incomplete*
- *Less recovery*
- *Poor material of construction*
- *More pollution*

**DISTILLATION UNIT OF CONVENTIONAL DESIGN**

**BEING EXTENSIVELY USED BY THE LOCAL FARMERS OF WESTERN UP IN INDIA**

CCRP  
Central Council for Research in Plantation Crops & Horticulture

**CONVENTIONAL RURAL DISTILLATION UNIT IN WESTERN U.P. AREAS OF INDIA**

CCRP  
Central Council for Research in Plantation Crops & Horticulture



**COIL  
CONDENSER**



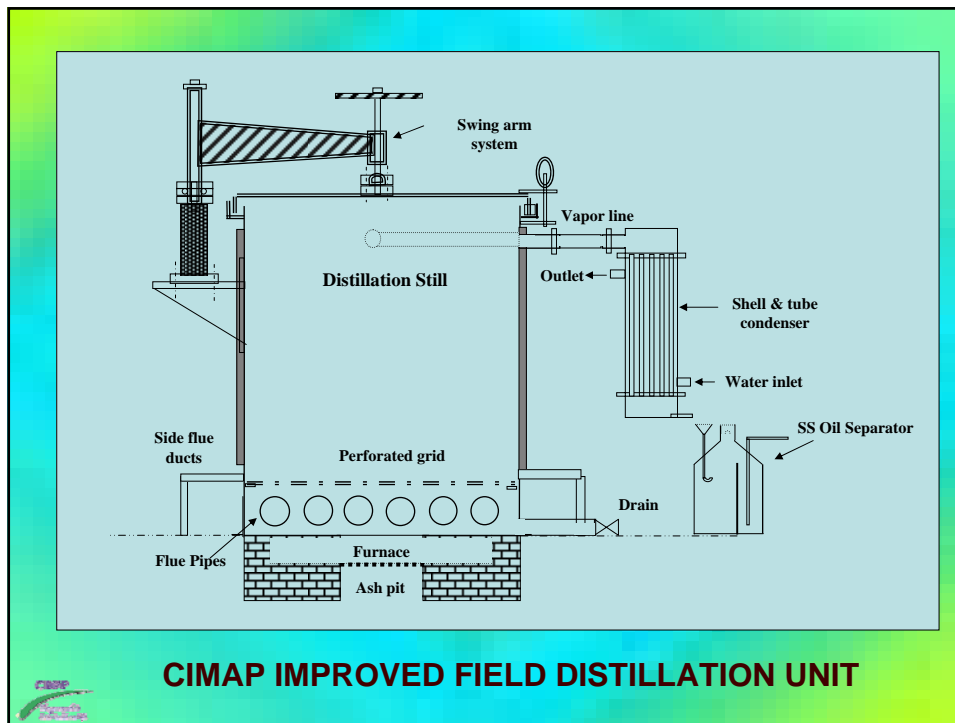
**DISTILLATION  
TANK**



**OIL SEPARATOR**

### **DESIGN IMPROVEMENTS IN DIRECTLY FIRED TYPES FDU'S**

- **HEATING AREA INCREASED BY ADDING FLUE PIPES.**
- **FURNACE RE DESIGNED WITH AIR CONTROL**
- **OIL SEPARATOR REDESIGNED WITH BAFFLE**
- **SHELL & TUBE CONDENSER FOR BETTER HTA**
- **CHIMNEY TO PREVENT SMOKE POLLUTION.**
- **SWING ARM ON LID TO EASE OPERATION**
- **IMPROVEMENTS IN M.O.C.**

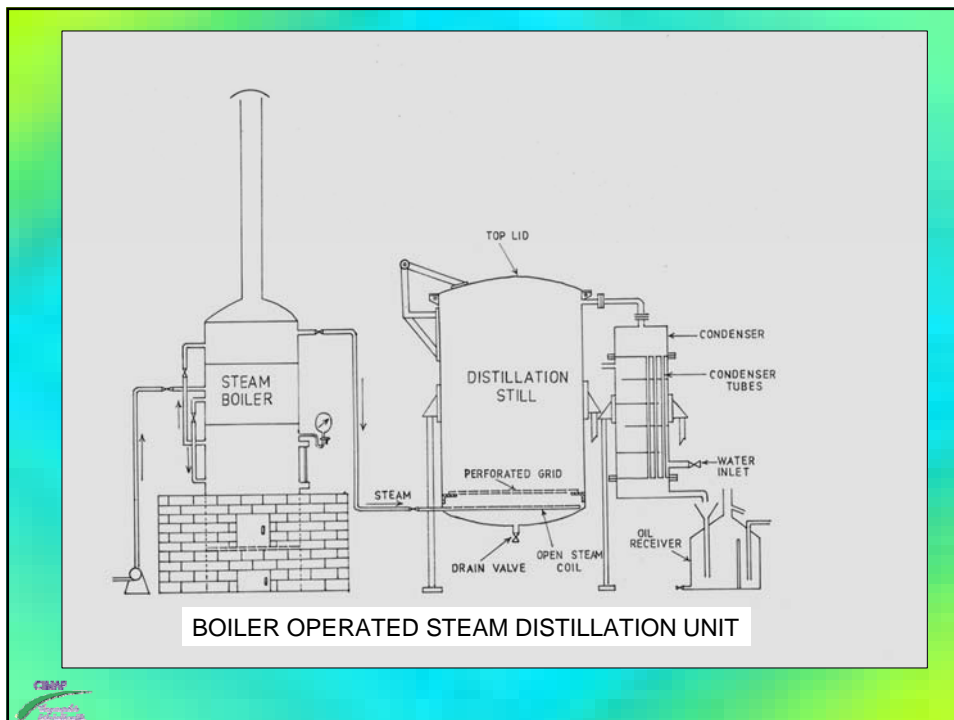


## ADVANTAGES OF IMPROVED FDU

- Higher steam generation as more heating surface area
- Better oil recovery, 10-15% higher
- Lesser time for distillation
- More fuel efficient, 20-30% fuel saving
- Utilizes agro waste / spent marc as fuel
- No smoke in work area
- Efficiently & technically designed
- Better quality material of construction
- Long life

## DIRECT STEAM DISTILLATION

- ⇒ Also referred sometimes as dry steam distillation
- ⇒ Plant material is supported on the grid and saturated steam from outside source ie Boiler is injected through steam coil
- ⇒ Steam in a field distillation unit is at atmospheric pressure and hence its temperature can be maximum 100°C. But steam in a modern pressure boiler operating at say 40 pounds per square inch pressure will have a temperature correspondingly higher.
- ⇒ The use of high pressure steam in modern steam distillation units permits much more rapid and complete distillation of essential oils.



## DIRECT STEAM DISTILLATION.....

- ⇒ Fuel costs are generally lower in modern steam distillation units due to higher thermal efficiency at which most of the boilers operate.
- ⇒ **Advantages : Steam control, no thermal decomposition and suitable for large scale and multiple units**
- ⇒ Capital cost is quite high so that only bigger producers can afford to own such units.
- ⇒ Still capacities range from 1 ton to 3 ton plant material per batch.
- ⇒ Material of construction could be mild steel, or stainless steel depending upon the corrosive nature of the essential oil.

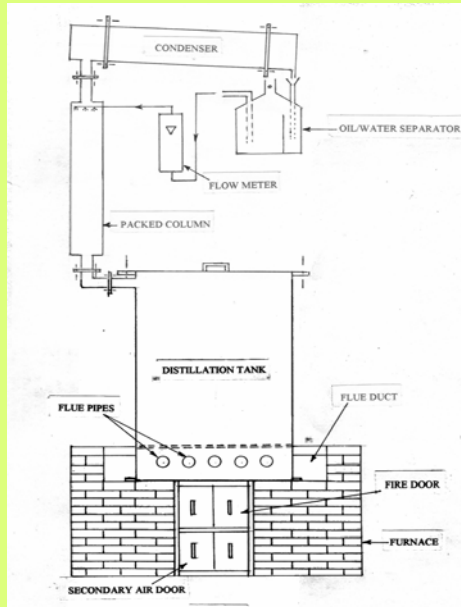


## **DISTILLATION WITH COHOBATION**

- ⇒ **Most essential oils have finite solubility in water but in certain oils like the oil of rose the solubility is quite high**
- ⇒ **In such a situation the loss of oil taking place with the outgoing waters of distillation can become alarmingly high**
- ⇒ **This problem is solved by returning the condensed water from the separator back to the still.**
- ⇒ **It cannot be done with steam distillation as the water level in the still will keep on building up due to continuous steam injection.**
- ⇒ **Instead the distillation is carried out in the mode of water and steam distillation**

## **DISTILLATION WITH COHOBATION.....**

- ⇒ **Condenser is moved above the distillation still so that condensed water from separator can flow by means of gravity to the still.**
- ⇒ **By limiting the total quantity of water in this closed cycle operation, it is possible to obtain increased yields of essential oils which are more water soluble**
- ⇒ **Cohobation of distillation waters in separate cohobation towers is quite widespread in Soviet Union and Bulgaria**



**DISTILLATION WITH COHOBATION**



## DESIGNING A DISTILLATION UNIT

*Main equipments of a Distillation unit.....*

- ➔ **Furnace ( Directly fired type units)**
- ➔ **Distillation Tank**
- ➔ **Condenser**
- ➔ **Oil Separator**
- ➔ **Boiler ( Steam Distillation units)**

## THE FURNACE

- ⇒ **Furnaces are used in directly fired type or more commonly known as the field distillation units**
- ⇒ **Designing of a furnace is important for optimum utilization of the fuel, maximum heat generation area and proper air flow control**
- ⇒ **The furnace should have separate heating /firing chamber with a fire grate and appropriately sized ash pit**
- ⇒ **The fire chamber should have optimum space for air inlet for maximum combustion of the fuel**
- ⇒ **The flue ducts should be designed for maximal heat transfer area to the tank**
- ⇒ **Chimney and furnace should be designed for maximum draft and proper complete firing**
- ⇒ **Fire doors should be provided in the both chambers to control the draft and firing**





## **THE DISTILLATION TANK**

- ❑ Cylindrical tanks are more preferred over square tanks
- ❑ Height to diameter ratio of tank still is an important design variable
- ❑ Most of the commercial stills are designed with a ratio of 1 to 1.5.
- ❑ Ratios lower than 1 result in uneven distribution of steam over the whole cross section of the tank whereas a ratio exceeding 1.5 may cause steam oil vapour to reflux back into the tank due to excessive tank height.
- ❑ Taller tank stills should be, preferably, insulated to overcome this problem.
- ❑ Choice of Material of construction of the tanks is vital for good quality oil
- ❑ Bulk density of the herbs to be distilled should be known before designing of tank

## THE DISTILLATION TANK.....

- ✘ In directly fired type modern distillation tanks, the integration of inbuilt calandria enhances steam generation rates
- ✘ Proper location and design of steam sparger in the tank bottom in boiler operated units is important to ensure even distribution of steam
- ✘ Tank walls should be fabricated with adequate metal thickness so that they do not buckle while transporting and erecting the still.
- ✘ Top lid of the still may be closed either with a metal flange or a water seal system of optimal height may be used.
- ✘ Discharging of spent plant material may be done from the discharge port provide near the still bottom, or alternatively the grid bottom may be lifted out with chain pulley hoist along with the spent plant material.



## Packed bulk density of some aromatic plants

Sl. No.	Plant material	Whole/Chopped	Hrs. Of Drying in Shade	Bulk Density kg/m <sup>3</sup>
1	Citronella java	Whole	Fresh	272
2	Citronella java	Chopped	Fresh	310
3	Citronella java	Whole	24 hrs	202
4	Lemongrass	Whole	Fresh	224
5	Lemongrass	Chopped	Fresh	308
6	Palmarosa	Whole	Fresh	246
7	Celery Seed	Whole	Air Dry	510
8	Celery Seed	Crushed	Air Dry	560
9	Vetiver	Whole	Air Dry	105
10	Vetiver	Chopped	Air Dry	130

\*Data obtained at CIMAP, India

## THE CONDENSER

- Two designs of condensers are most popular in the essential oil industry. The older submerged coil type and the more modern shell and tube type.
- Optimum design of condenser is critical because an undersized condenser will allow steam – oil vapors to escape uncondensed and over sizing the condenser means unnecessary extra capital cost.
- Heat removal capacity of a condenser is expressed by the following equation

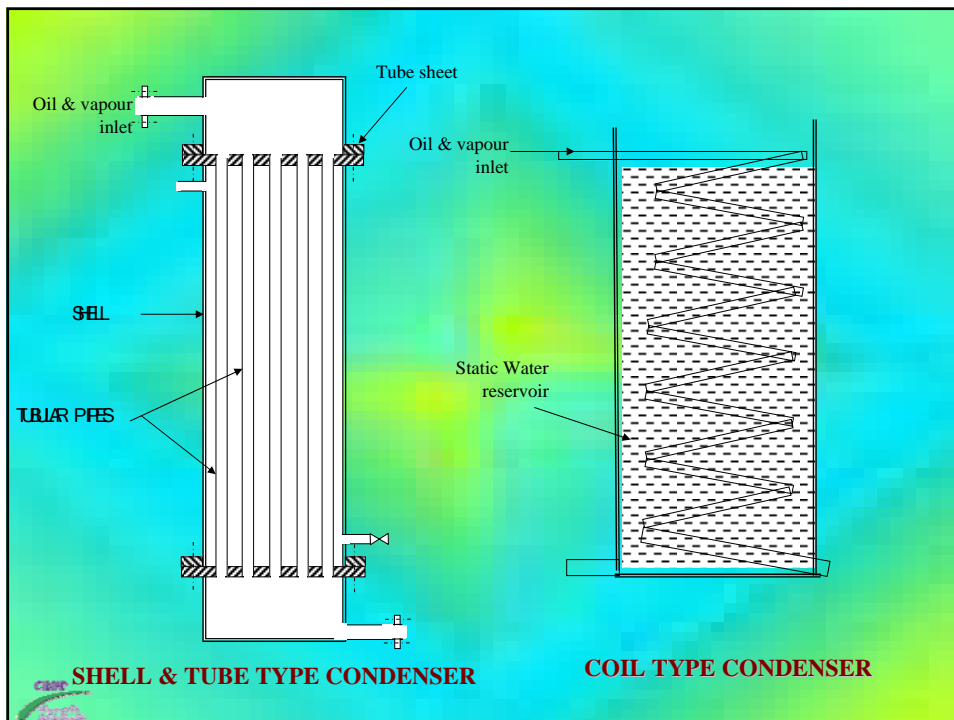
$$Q = U \times A \times T$$

Where Q = Heat removal rate (BTU/hr)

U = Heat transfer co-efficient of condenser expressed as;  
BTU/hr Ft<sup>2</sup>0F)

A = Area available for heat transfer; (Sq ft.)

T = Log mean temperature difference between cooling water  
and condensate, (°F)

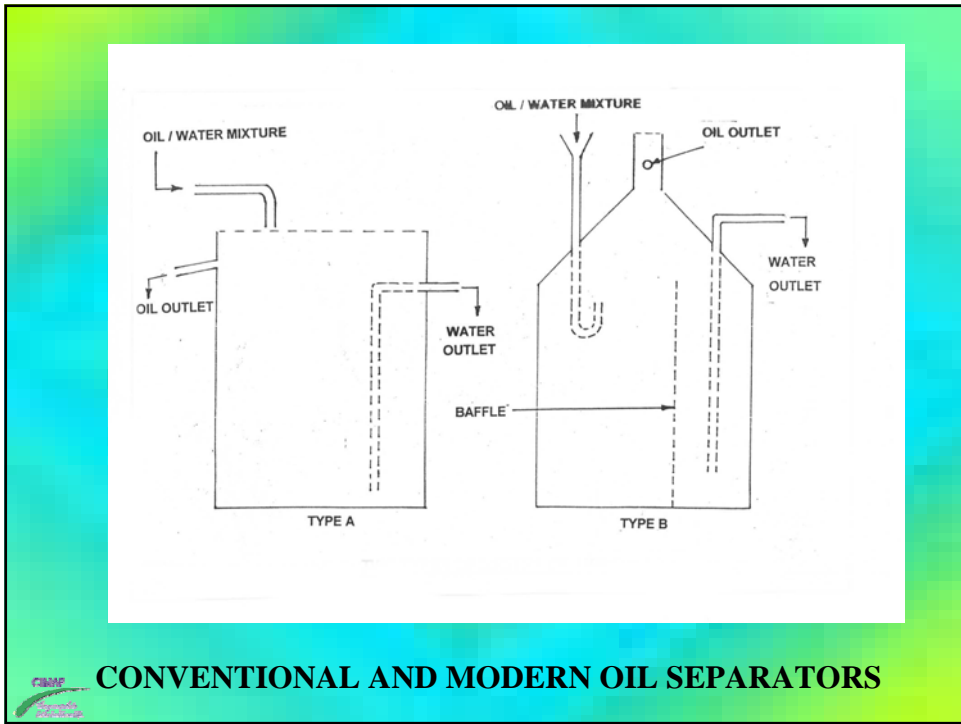
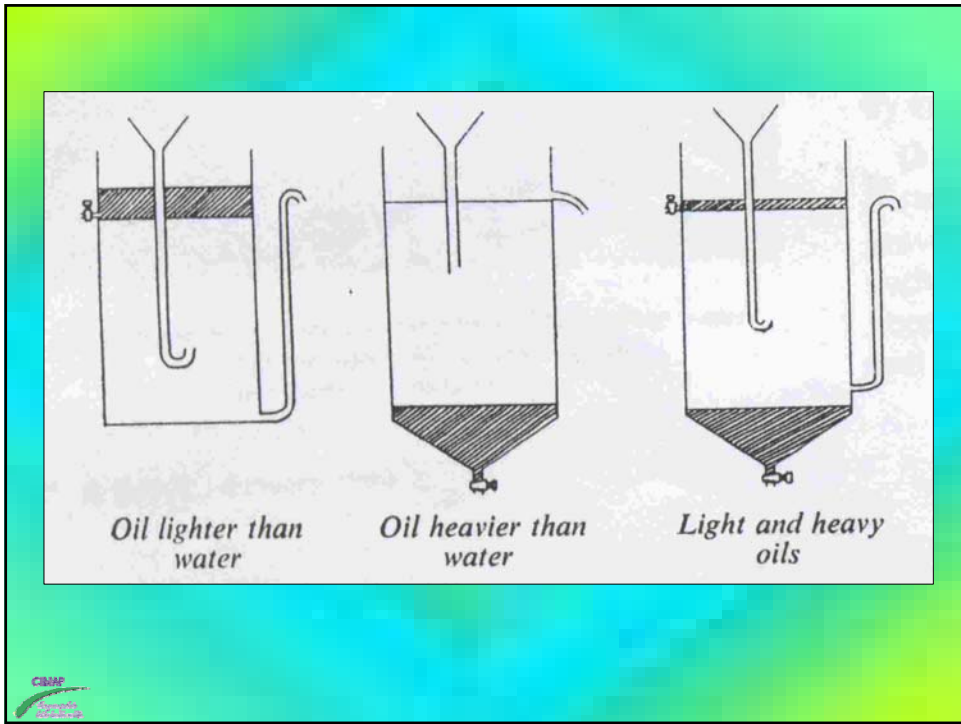


## COIL AND SHELL & TUBE TYPE CONDENSERS

COIL TYPE CONDENSERS	SHELL & TUBE TYPE CONDENSERS
Easy to fabricate	Requires technical designing and difficult to fabricate
Low cost	High in cost
Generally fabricated using MS / GI pipes which deteriorates quality of oil	Fabricated using Stainless steel tubes generally with MS shell
Poor heat transfer due to less heat transfer area and low heat transfer coefficient	Good heat transfer due to high heat transfer area and higher heat transfer coefficient
Not safe as risk of blockage high resulting in high pressure build ups in tanks	Safe as multiple tubes ensures no blockage
Requirement of water tank and regular replacements of water	Requires continuous running water supply

## THE OIL SEPARATOR

- Oil separator has to perform the crucial function of separating the essential oil from the condensed steam.
- Generally oil is allowed to accumulate in the vessel, to be drawn off periodically, whereas the condensed water flows out continuously
- A great variety of designs for separators are in use depending on oil density
- There is an optimum temperature of the condensate at which the oil- water separation is most complete
- Oil separator must have sufficient holding volume so that the entering oil – water mixture gets enough time to separate i.e the residence time.
- Installation of a baffle barrier before the water outlet considerably improves separation of oil.



**CONVENTIONAL AND MODERN OIL SEPARATORS**

## THE STEAM BOILER

- Availability of the kind of fuel will generally determine the choice of the type of boiler.
- Oil fired boilers are very convenient to operate and start up. They are most amenable to automate instrumentation and require minimum supervision.
- Coal and fuel wood fired boilers are simpler in construction, take longer to start up and require manual operation and supervision.
- Many of these boilers can be converted for firing with locally available waste materials like rice husk and spent essential oil plant material.
- Capacity of the boiler will depend on the size and number of distillation units

## Relative costs of Boiler fuel in India

Fuel	Cost Rs/kg	Heating Value kcal/kg	Cost of 1,000 kcal (Paise)
L.D.O.	35.0	10,700	327
Furnace oil	20.0	10,600	141
Steam coal	5.0	7,900	63
Fire wood	2.0	4,700	43
Rice husk	0.6	3,300	18
Spent Citronella Grass	0.15	3,300	5

## PARAMETERS EFFECTING YIELD & QUALITY OF ESSENTIAL OILS

### »»> Mode of Distillation

*Technique for the distillation should be chosen on basis of oil boiling point and nature of herb as the heat content and temperature of steam can alter the distillation characteristics*

### »»> Proper design of equipment's

*Improper designing of tank, condenser or separators can lead to loss of oils and high capital investments*

### »»> Material of Construction of equipment's

*Essential oils which are corrosive in nature should be preferably distilled in stills made of resistant materials like aluminium, copper or stainless steel*

### »»> Condition of Raw material

*Condition of raw material is important because some materials like roots and seeds will not yield essential oil easily if distilled in their natural state. These materials have to be crushed, powdered or soaked in water to expose their oil cells.*



### »»> Filling of raw material / steam distribution

*Improper loading of the herb may result in steam channeling causing incomplete distillation*

### »»> Operating parameters like steam injection rate inlet pressure/ condensate temperature

*Proper control of injection rates and pressure in boiler operated units is necessary to optimize the temperature of extraction for maximal yield*

*Temperature of condensate should not be high as can result in oil loss due to evaporation*

### »»> Time given for distillation

*Different constituents of the essential oil get distilled in the order of their boiling points. Thus the highest boiling fractions will be last to come over when, generally, very little oil is distilling. If the distillation is terminated too soon, the high boiling constituents will be lost.*

### »»> Pre condition of tank / equipments

*Tanks should be well steamed for multiple crop distillation*

*Tank / equipments should not be rusted for quality oil*



### EFFECT OF STEAM PRESSURE ON YIELD OF ESSENTIAL OILS

S NO	RAW MATERIAL	STEAM PRESSURE (PSIG)	OIL %
1	Cedarwood	19	4.18
2	-do-	34	4.41
3	-do-	50	3.67
4	Nagarmotha	0	0.32
5	-do-	20-25	0.50

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### EFFECT OF DRYING *M. ARVENSIS* HERB ON YIELD OF ESSENTIAL OIL.

Period of Drying (DAYS)	Shade drying		Sun drying	
	% Moisture	Oil %	% Moisture	Oil %
0	69.0	0.84	69.0	0.84
1	46.5	0.88	29.5	0.82
2	23.3	0.90	15.3	0.81
3	18.5	0.84	6.85	0.80
4	12.5	0.83	3.5	0.79
5	8.5	0.81	1.0	0.75
6	6.0	0.80	Nil	0.70
7	4.0	0.79	Nil	0.66

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### EFFECT OF MODE OF DISTILLATION ON YIELD OF ESSENTIAL OILS

Raw Material	Yield of essential oil (%)		
	Hydro- Dist.	Water & Steam distillation	Steam distillation
Vetiver Roots	0.25	0.25	0.08
Nagarmotha	0.20	0.32	0.41
Celery Seed	1.35	-	0.80
Cedarwood	3.7	-	4.40

### Effect of Raw material size on yield of essential oil

S. No.	Raw Material	Size	% oil yield
1.	Palmarosa	Whole herb	0.49
2.	-do-	Chopped	0.55
3.	<i>Mentha piperita</i>	Whole herb	0.47
4.	-do-	Chopped	0.44
5.	Nagarmotha	Whole herb	0.08
6.	-do-	(-6) Mesh	0.40
7.	-do-	(-12) Mesh	0.35
8.	Lemongrass	Whole herb	0.33
9.	-do-	Chopped	0.34

## TREATMENT / PURIFICATION OF ESSENTIAL OILS

- ❖ Essential oil as obtained from the oil separator is in crude form.
- ❖ It may have suspended impurities and appreciable moisture content.
- ❖ It might even contain some objectionable constituents which degrade its flavour / fragrance quality.
- ❖ The presence of moisture and impurities adversely affects the keeping quality of oil and accelerates the polymerization and other undesirable reactions.

### *Remedies*

- ❖ Filtration of oil through Marlin is a simple method for removal of impurities
- ❖ For removal of the moisture and free the oil of suspended impurities, addition of a drying agent like Anhydrous Sodium Sulphate to the oil, standing the oil overnight will get the oil clear of moisture
- ❖ On industrial scale use of high speed centrifuge to clarify the essential oils can be also used.
- ❖ Essential oils can also be rectified or re-distilled to remove objectionable constituents, dark colour or polymerized oil

## STORAGE OF ESSENTIAL OILS

- Oils should be stored in shaded areas away from direct sunlight
- Should always be filled up to brim level
- Containers / bottles should be well cleaned / steamed

CONTAINER MATERIAL	REMARKS
G.I. ( White Sheet)	For all oils
Aluminum	For all / high value oils
Iron / Mild Steel	For some oils. Not recommended for long storage
Stainless Steel	For all high value oils
GI / MS with glass/Stainless Steel lining	For high value oils
Plastic / HDPE	For all oils, for less time only
Glass Bottles	For all oils, lesser quantity only

## *"CIM-ASAVIKA"*

### **CIMAP MULTI UTILITY PORTABLE DISTILLATION UNIT**

- **LOW COST PORTABLE TYPE DISTILLATION UNIT SUITABLE FOR SMALL ENTREPRENEURS AND RURAL FARMERS**
- **PURE STAINLESS STEEL CONSTRUCTION WITH LONG LIFE**
- **SHELL & TUBE CONDENSER FOR PROPER CONDENSATION**
- **CAN BE OPERATED BY FIREWOOD, AGRO WASTES, LPG / KEROSENE BURNERS**
- **MODULAR CONSTRUCTION FOR EASY ASSEMBLY AT SITE**
- **CAN BE OPERATED EVEN BY LAYMAN AND WOMEN**
- **NO POLLUTION AT WORKPLACE DUE TO PROVISION OF CHIMNEY**

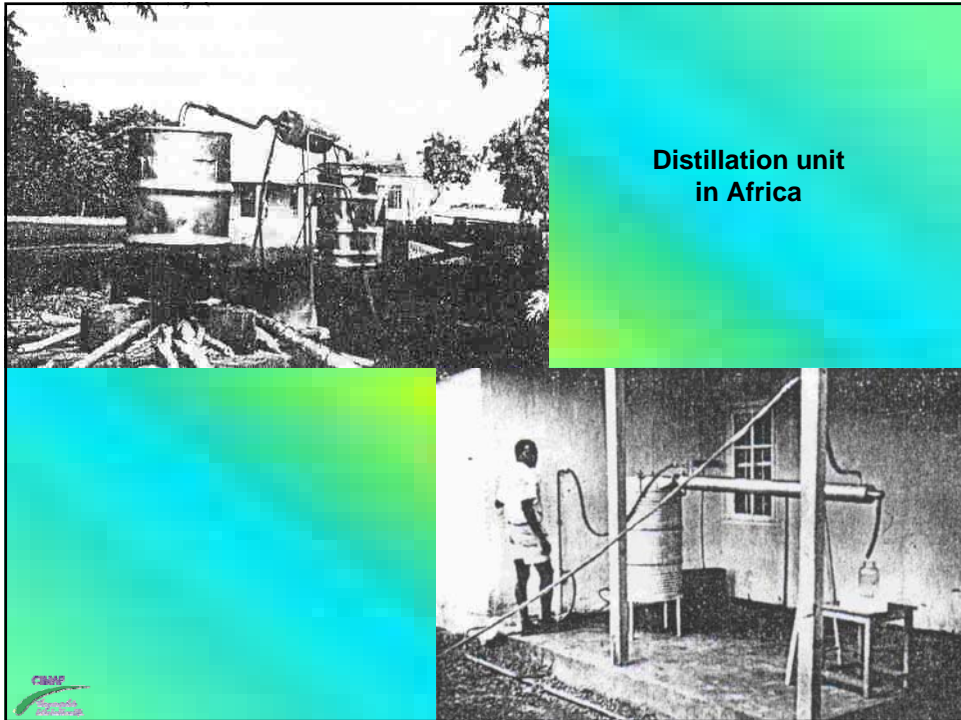


**Some glimpses of different distillation units.....**













**MOBILE DISTILLATION UNIT**

THANKYOU





- **ESSENTIAL OILS : BASICALLY A COMPLEX MIXTURES OF HUNDREDS OF MONOTERPENES, THEIR ALDEHYDES ALCOHOLS, ESTERS AND ACIDS ETC.**
- **THE FRAGRANCE THAT WE SENSE IS THE COMPOSITE EFFECT OF ALL THE CONSTITUENTS PRESENT IN IT.**
- **TRACE CONSTITUENTS PRESENT IN THE OIL <math><0.5\%</math> LEVEL PLAY VERY IMPORTANT ROLE IN IMPARTING THE CHARACTERISTIC LIFTING AND LASTING EFFECT**
- **PERFUMERS HAVE TRAINED SENSE OF SMELL AND GO BY JUST RUBBING A DROP OF OIL ON BACK OF PALM AND SMELLING**
- ***BUT THE QUALITY OF THE OIL CANNOT BE CONVEYED MERELY BY WORDS AND SHOULD NOT BE THE CRITERIA FOR THE JUDGMENT OF QUALITY OF THE OIL***
- ***TO OVER COME THIS PROBLEM SCIENTIFIC APPROACH WITH SOME SCIENTIFIC METHODS HAVE TO BE VALIDATED***

### **METHODS COMMONLY BEING EMPLOYED FOR QUALITY ANALYSIS OF OILS**

- **INSTRUMENTAL TECHNIQUES LIKE GC / GC MS FOR CHEMICAL FINGERPRINTING / IDENTIFICATION & QUANTIFICATION OF CONSTITUENTS**
- **PHYSICO- CHEMICAL PROPERTIES LIKE RI, S.G, O.R, VISCOSITY, BP, CP, ACID NO, ESTER NO. ETC**
- **PERCEPTION BY THE SENSORY ORGANS I.E. OLFACTORY AND ORGANOLPETIC PROPERTIES LIKE ODOUR AND TASTE**

### **KEY REASONS FOR VARIATION IN QUALITY OF THE OILS**

- **VARIETY / CULTIVATION / HARVESTING TIME or TECHNIQUES**
- **IMPROPER DISTILLATION / STORAGE**
- **ADULTERATION**

### MAIN TYPES OF ADULTERATION

- **INVISIBLE : THOSE MATERIALS UNDETECTABLE BY GC ; EG VEGETABLE OIL/ MINERAL OILS LIKE RAPE SEED OIL WHICH CAN BE DETECTED BY SOLUBILITY TESTS AND CHEMICAL DERIVITISATION**
- **VISIBLE : NORMALLY DETECTABLE BY GC : INCLUDES A NUMBER OF SOLVENTS & PERFUMERY MATERIALS EG DIPROPYLENE GLYCOL (DPG) AND TRYPROPYLENE GLYCOL METHYL ETHER (TPGME) - PHTHALATE ESTERS SUCH AS DIBUTYLPHTHALATE (DBP), DIOCTYL PHTHALATE (DOP) ETC. CAN BE DETECTED BY EXPERT ANALYSTS BY INSTRUMENTAL TECHNIQUES**
- ⊗ **IN ALL INSTANCES OF VISIBLE AND INVISIBLE ADULTERANTS THE ADDED MATERIAL IS NEARLY A DILUENTS AND MAKES NO ODOUR CONTRIBUTION OF ITS OWN.**
- ⊗ **ADDITION OF 10- 15% OF SUCH A MATERIAL MAY PASS UNNOTICED IF THE MATERIAL IS EVALUATED AGAINST ON A ODOUR BASIS, EVEN BY AN EXPERT NOSE.**
- ⊗ **BUT CAN BE DETECTED BY MODERN INSTRUMENTS AND PHYSICOCHEMICAL EVALUATIONS**