

09/10/19

# Unit - 5<sup>th</sup>

## Extraction

\* Extraction is defined as process which is used to separate the phytoconstituent from the insoluble matrix of the plant.

\* It is of two type

i) Extraction of solid from the solid part of material.

ii) Solvent extraction which is used to extract the liquid from the liquid.

\* There are several factors which affect the extraction procedure.

1) The nature of extracting solvent  $\Rightarrow$  based on the concept of like dissolve like polar material can be extracted from polar solvent while non-polar material will be extracted from the non-polar solvent.

2) The plant particle size  $\Rightarrow$

should be in powder form as it will reduce the particle size & provide The drug

the better penetration of the solvent & also the every particle of the plant material will be in contact with in the solvent.

3) Solvent to plant material ratio  $\Rightarrow$  Fresh solvent must be introduced after a particular time as excessive amount of solvent leads to the high cost and it will also decrease the extraction efficacy.

4) Temperature  $\Rightarrow$  Temperature must be controlled during the extraction procedure at high temp. may cause the loss of solvent.

5) Menstruum  $\Rightarrow$  It is the solvent which is used for extraction and to dissolve the active medicament present inside the plant & animal tissues.

6) Marc  $\Rightarrow$  It is an inert insoluble material left after exhaustion of the plant material.



## 2) Conventional method of Extraction ⇒

### Infusion ⇒

It is the procedure in which the crude drug and the vegetable crude drug it introduce in the boiled water and the then solution allowed to cooled & filter for the administration.

### Note ⇒

The infusion to be administered should be freshly prepared.

Vegetable Crude drug + Boiled water (menstruum)



Cool



Filter

### Concentrated Infusion ⇒

In this alcohol is used as a menstruum

### Decoction ⇒

It is the process in which the hard crude drug is mixed with the water and allowed to boiled for some time and

then the mixture is allowed to cool  
filter it and dispense.

Hard crude drug + water  
↓ Boil  
Cool  
↓  
Filter  
↓  
Dispense.

21/10/19

## Digestion

In this method gentle heat is applied over the substance to be extracted as it will help the solvent to dissolve more drug from the substance.

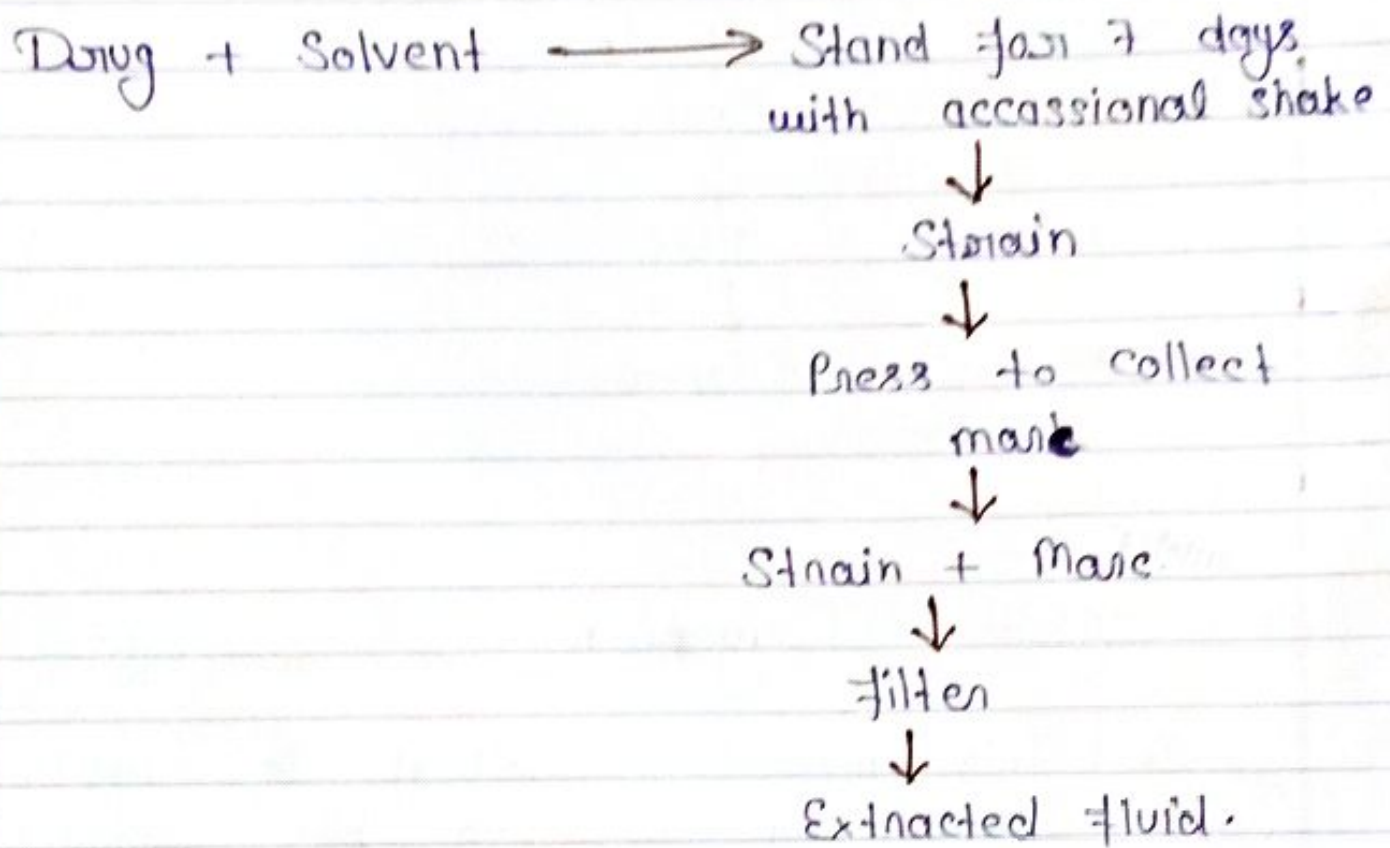
## Maceration

The drug sample is mixed with the menstruum and allowed to stand for 7 days with occasional shaking and after 7 days the mixture is being strain by pressing to collect the remaining marc both the filtrated are mixed to collect the



result in solution.

This process is used for preparing the tincture



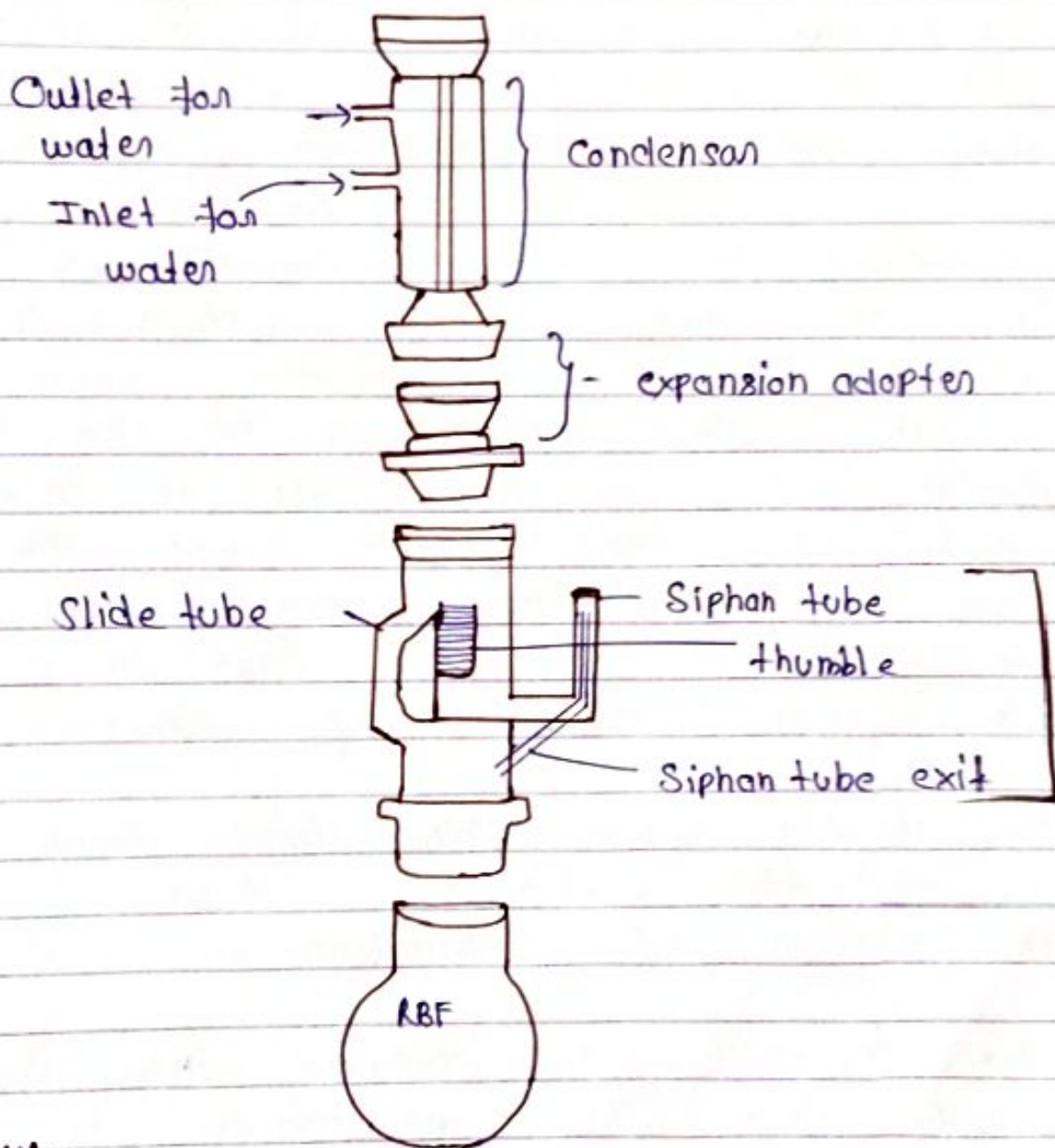
### i) Double & Triple Maceration

Double maceration is used to prepared the conc. infusion while triple maceration is used in those cases where the sample can not be pressed to collect the infusion as in case of Ligustice.

## Soxhlet Extraction

Temp. 70-90°C

It is invented by Franz von Soxhlet in 1879 for the extraction of lipid.



23/10/19

- The material to be extracted is finely coarsely powder and packed inside in the thimble by packing inside the



Filter paper is covered by cotton. From the cotton top in order to avoid the <sup>entry</sup> ~~entry~~ of drug particle in the siphon tube.

- Thimble any siphon tube is called as the siphon extraction which is attached to the round bottom flask from the condenser on top from ↓ menstrum is the extract added slowly once the solvent is list to the top of the siphon tube it will revert back to the round bottom flask which is attached to the heating mantle the condenser has an inlet & outlet for the water once the solvent begins to boil the vapor travel to the condenser and fall back in a thimble in the form of hard liquid.
- The thimble containing drug begins the process of extracting solvent & begins the process of extraction.
- The solvent get emptied into the round bottom flask the process will repeat for several cycles till the process procedure get completed.

### Advantage →

- This process is automatic & continuous.
- Process is time saving and cost effective.

### Disadvantage →

- In this process the extracted phytoconstituent will boil continuously with the solvent which may affect the quality of the extracted phytochemical therefore this method is not suitable for thermolabile phytoconstituents.

## Percolation

It is the most common method for the preparation of a tincture and fluid extract.

It involves three steps first-

Imbibition

Maceration

Percolation

### 1) Imbibition →

In this process the plant material is introduced into the percolator and allowed to stand for four or



hours during this process the plant material will get swell and solvent will get inside the powder sample.

## 2) Maceration ⇒

During this process and extra solvent is being added to the previous sample and allowed to remain for 24 hours with occasional shaking this causes saturation of the powder sample with the solvent.

## 3. Percolation ⇒

It allowed the saturated menstrum to settle down and drug is collected from the column.

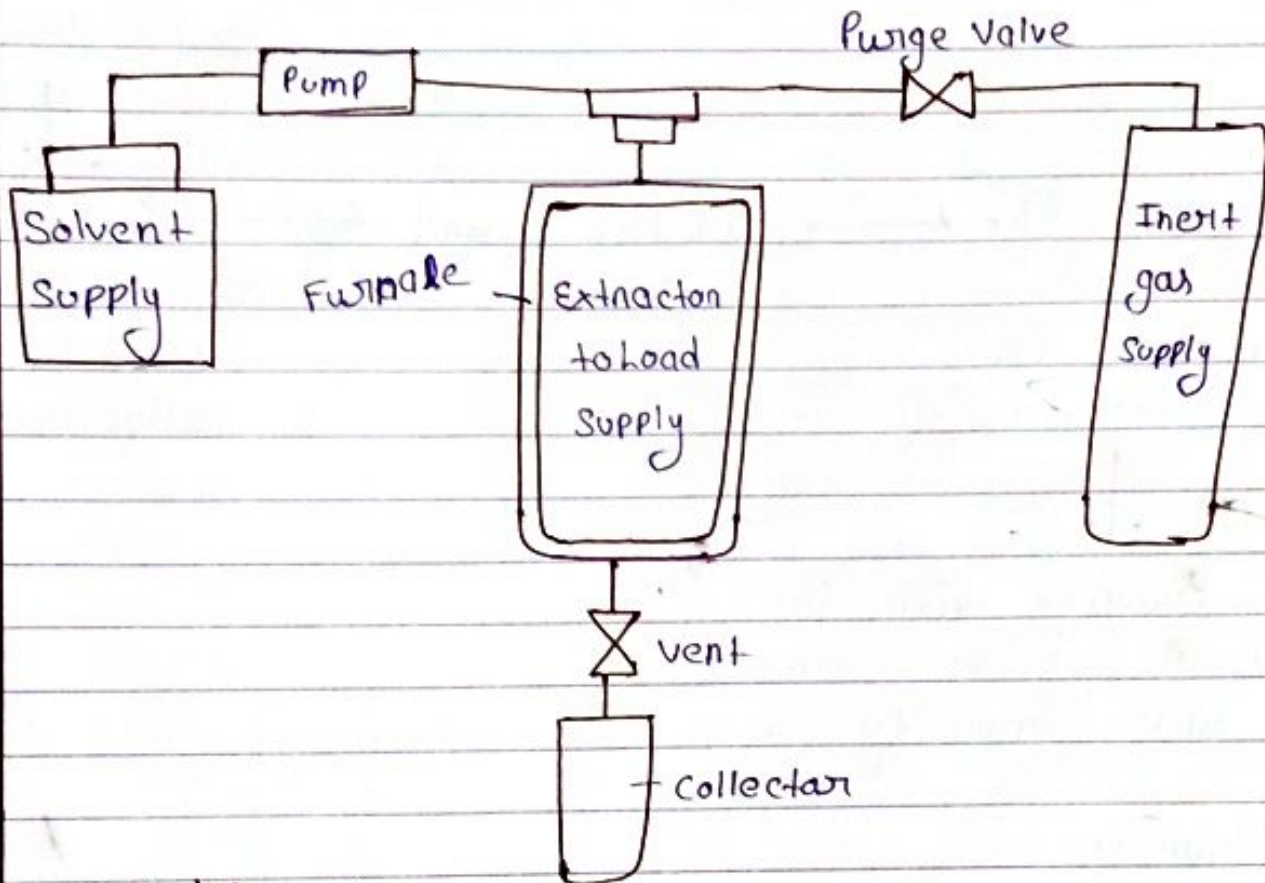
For the percolation, percolator is used which is conical in shape and having opening at the top and adjustable column in the bottom to allow passage of fluid at convenient rate.

## Pressurized Fluid Extraction

It is also known as accelerated solvent extraction or high pressure solvent extraction.

This process is carried out at high pressure.

to accelerate the extraction process and high pressure.



30/10/19

The solvent is filled into the solvent supply chamber and sample is put into the extraction chamber. Cool temp. maintain to  $0$  to  $100^{\circ}\text{C}$  & pressure is maintain about  $10$  to  $20$  Pascal for some time the extracted material is collected into the collection vial & allow the second solvent to run and collect the extracted material in collection vial by purging it with the inert gas to remove any impurity & to avoid lump formation.



Sample in extraction cell  $\longrightarrow$  Solvent from solvent supply  $\longrightarrow$  Temp. 30 to 100°  
& pressure 10 to 20

Collect the extracted material in collection vial  $\longleftarrow$  Run the second solvent  $\longleftarrow$  collect the extracted material in collection vial

$\downarrow$   
Purging with the inert gas to remove any impurity.

Advantage :-

- It can be used with the less quantity of sample.
- Process is fast & can save the time.
- It is suitable with the phytoconstituent available in less quantity.
- It is cost effective.

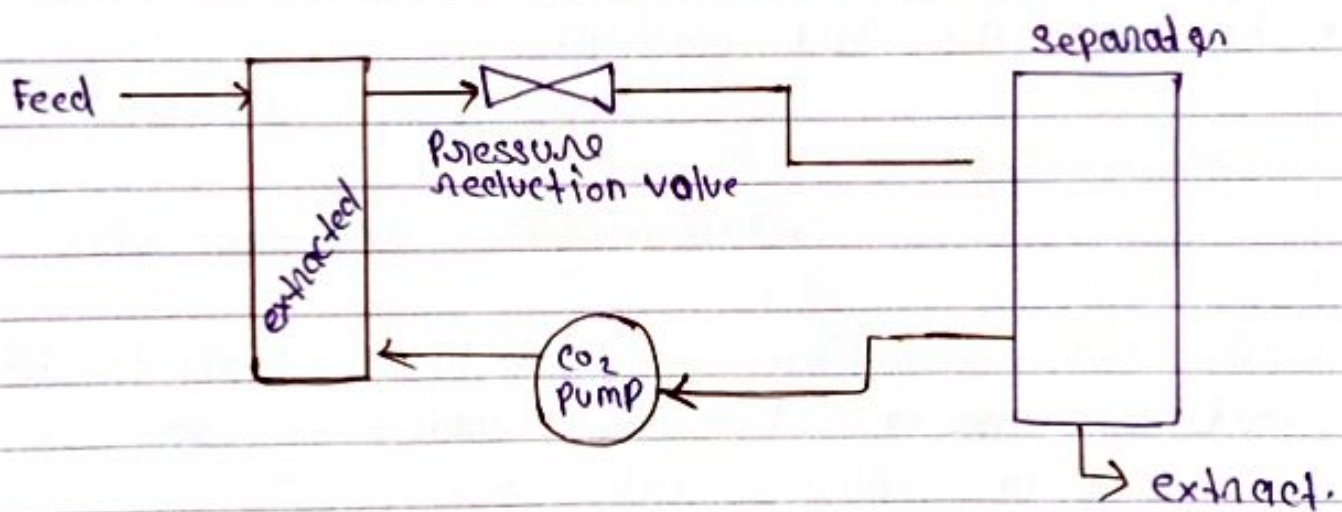
Dis-advantage  $\Rightarrow$

It is not suitable for the thermolabile substance.

## Super Critical Fluid extraction

Super critical fluid are those fluid where the liquid & upper phase of a substance is indistinguishable at a certain pressure & temp. that is these fluid contain the property like the liquid phase & gas phase.

Use: The process is used to remove any unwanted entities from the product that is during the decaffeination process.



The material to be extracted is feed into the extractor along with the solvent gets in contact with the  $\text{CO}_2$  from  $\text{CO}_2$  pump with high pressure (about 380 pascal) the extracted phytoconstituent is sent to the separator with the low pressure (150-180 pascal) through the pressure reduction valve the extracted material is collected while the



$\text{CO}_2$  is again sent back to the  $\text{CO}_2$  pump.

This is the semibatch process as the flow of  $\text{CO}_2$  is continuous while the material needs to be extracted is feed into the batches in the extractor.

Advantage  $\Rightarrow$

- It provides an advantage of selecting the pressure as the pressure too is used to extract.
- This is the fast process.

Disadvantage  $\Rightarrow$

- Operational cost is very high.
- For the extraction of nonpolar phytoconstituent, modifiers need to be added due to the nonpolar nature of  $\text{CO}_2$ .

Use.

- It is used to extract ginger oil, Rosemari oil, sinnamal bark oil, nutmeg oil.
- For the decaffeination of tea & coffee.

31/10/19

## Spectroscopy $\Rightarrow$

It is the phenomena of absorption and emission of electromagnetic radiation by matter.

## UV visible spectroscopy $\Rightarrow$

It deals with the absorption of UV radiation the colourless compound absorbed the UV radiation in the range of 200 to 400 nm why while the coloured compound absorbed the visible radiation in the range of 400 - 800 nm.

## Application $\Rightarrow$

### Qualitative Analysis $\Rightarrow$

The graph is plotted with the absorption on y axis & wavelength on x-axis the graph is termed as the absorption curve the wavelength at which maximum absorption occurs known as  $\lambda_{max}$  It will remain unchanged by  $T_{es}$  on  $\downarrow$ es the conc. the colour substance absorbed radiation of different wave length.

### Quantitative Analysis $\Rightarrow$

It is the graph is plotted with the absorbance & conc. of a substance in sample solution



## Infrared of vibrational radiation

It is a technique in which infrared light is used to study its interaction with the molecules. The spectra generated during IR give the information about the functional group of the compound.

### Application -

It is used to identify the functional group and to elucidate the structure of the compound.

It is also used to determine impurities in the compound as IR gives additional peaks due to the presence of impurities.

### Mask Spectroscopy - an Positive ion spect.

It is particularly used to determine the molecular weight of a compound. Mask spectroscopy is a result of ions produced in the form of a graph known as a mask spectrum.

Which is a plot of intensity vs. mass-to-charge ratio.

It does not require electromagnetic feed for excitation.

## Application ↵

- Structure elucidation
- Impurity detection.

## Electrophoresis

It is the movement of charge particle under the influence of electric field it is of two type.

1. Cataphoresis
2. Anaphoresis

### 1) Cataphoresis ⇒

It involves the movement of electron of +ve charged particle towards the -ve electrode under the influence of magnetic field.

### 2) Anaphoresis ⇒

It involves the movement of -ve charged electrons towards the +ve electrode.

A

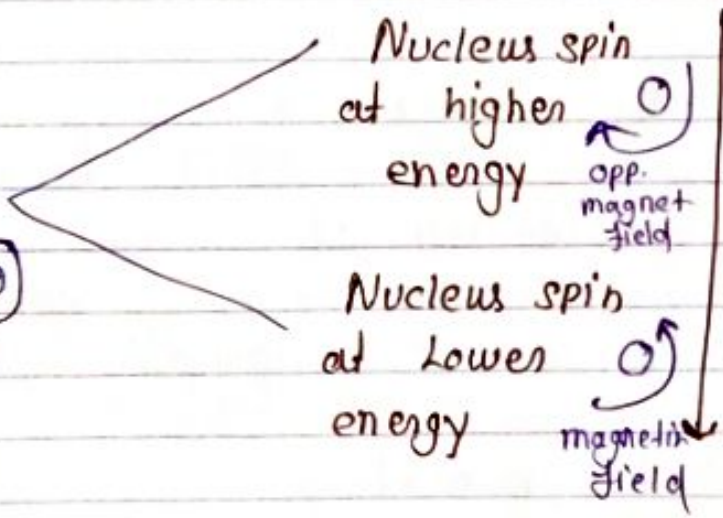


# NMR

The nuclei are electrically charged & have spin that cause them to behave like a magnet.

The nucleus having spin at higher energy generate a magnetizing field in opposite direction to external mag magnetizing field where as the nucleus the spin at lower energy generate a magnetizing field in the direction of external magnetizing field.

Nucleus Behaves as a magnet due to its spin rotation



external magnetic field.