Pharmacognosy & Phytochemistry-I

(B.P504T)

UNIT-V

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*Modern Methods of Extraction :-

① Ultrasound-Assisted Extraction / Sonication Extraction

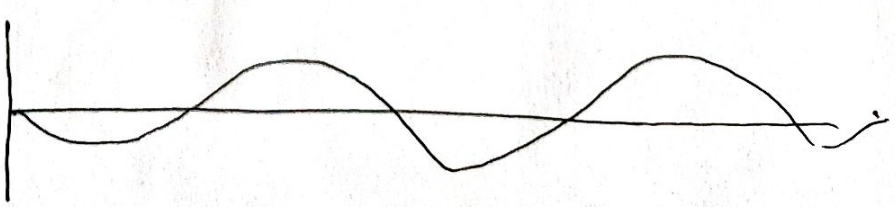
Ultrasound :- The term "ultrasound" (sound waves) applies to all acoustic energy with a frequency above human hearing (20,000 hertz or 20 KHz). The ultrasonic wave has mechanical vibrations with frequencies ranging from 20 KHz to 10 GHz. It is a non ionizing and non invasive source of mechanical energy.

- Sonication refers to the process of applying sound energy to agitate particle.

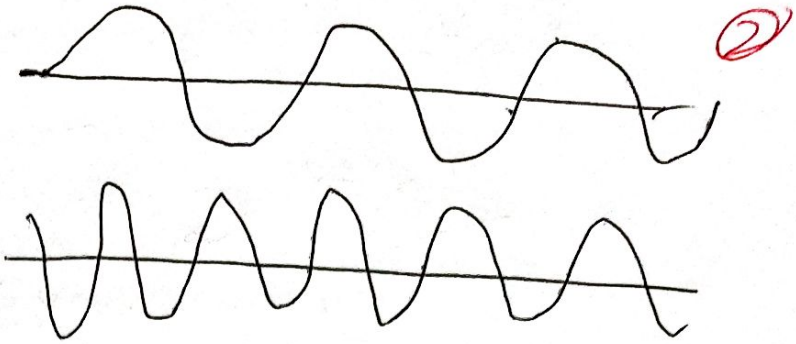
***UAE :-** In this extraction process, mechanical energy generated by ultrasound waves, is applied to the samples to accelerate the extraction. This technique is based on the application of high frequency sounds and a limited amount of solvent to produce an effective extraction of the compounds contained in a solid matrix.

~~Infra sound~~

Infrasound- (below 16 Hz)



Audible frequencies. (16 Hz - 20 kHz)
Ultrasound (over 20 kHz).



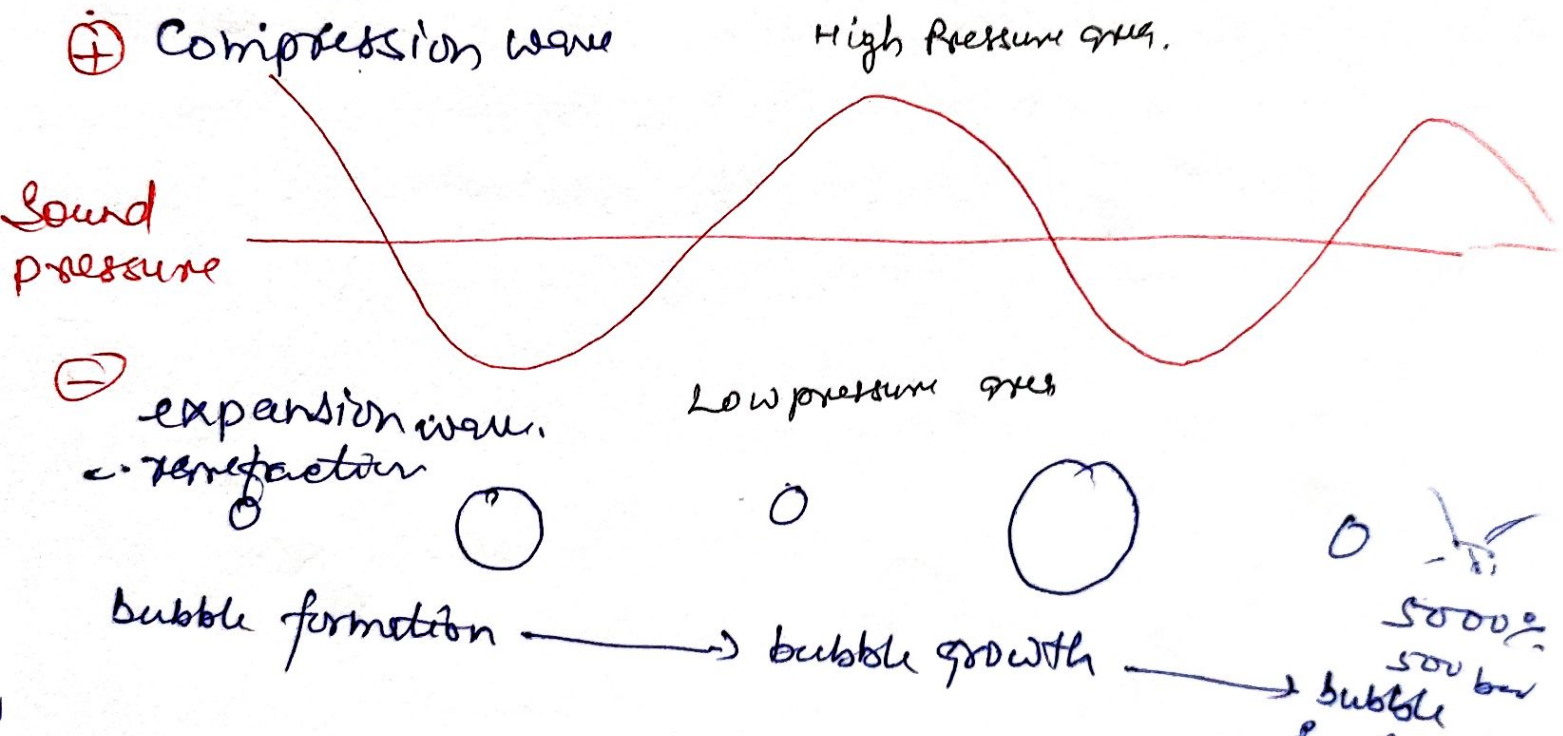
Principle of Ultrasound-Assisted Extraction (UAE) ⇒

The main driving force for the extraction effects of sonication is acoustic cavitation.

- When ultrasonic energy in the form of waves pass through a liquid solvent containing solid particles, it induces a series of compressions and rarefactions in the molecules of the medium.
- Such alternating pressure changes cause the formation and ultimately, the collapse of bubbles in a liquid medium.
- This phenomenon of creation, expansion and implosive collapse of microbubbles in ultrasound irradiated liquid is known as acoustic cavitation.
- When cavitation bubbles oscillate and collapse several physical effects are generated (eg - shock waves, microjet, turbulence).
- Implosion at the solid sample resulting in localized high temperature (about 4500°C) and pressure (50 MPa). Temperature and pressure changes that occur from these implosions cause ⟶

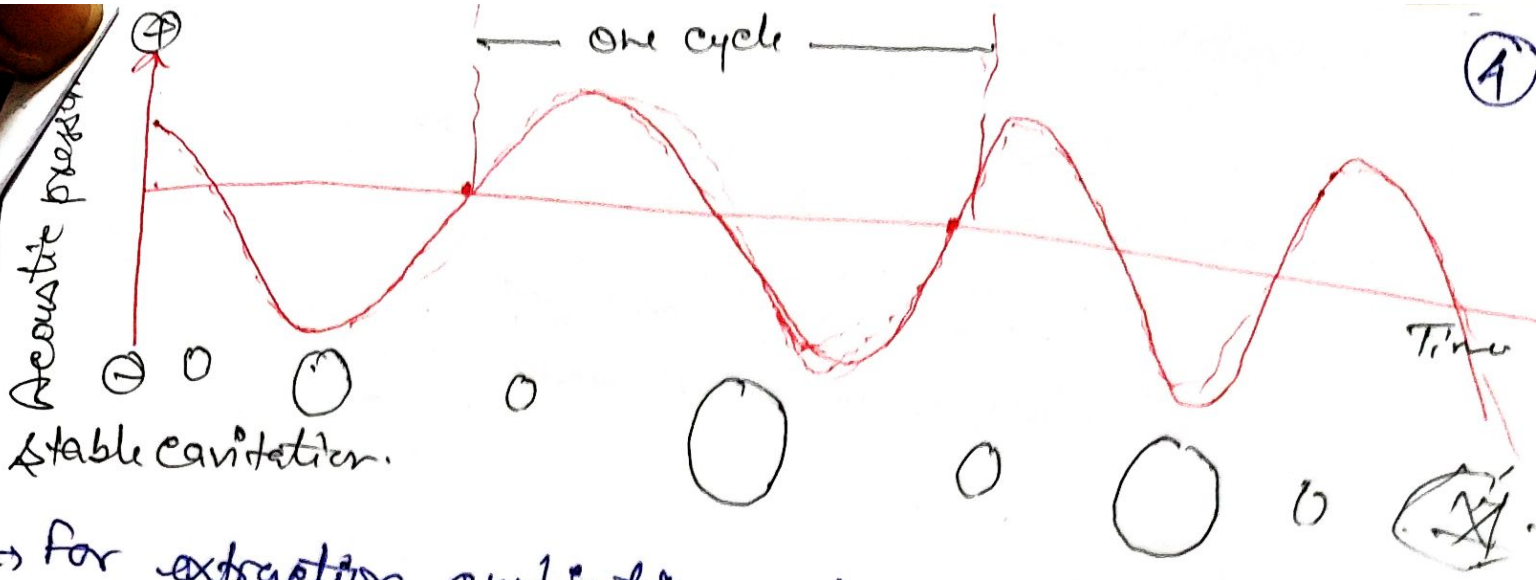
→ Increase mechanical stressing of the cells.
→ Thinning of cell membranes and cell disruption.
→ Increases the permeability of the cell walls.

⇒ Resulting in enhanced solvent penetration into cells and in this way the mass transfer rate and the solubility of the analytes (target compound) into the solvent are improved. Hence effective and fast extraction is possible.



⇒ Cavitation bubbles formed are divided into two types

1. Transient cavitation (inertial) – very short period sometime less than one cycle and collapse violently.
2. Stable cavitation (non inertial) – long-lived gas bubbles and exist for many cycle of compression and rarefaction



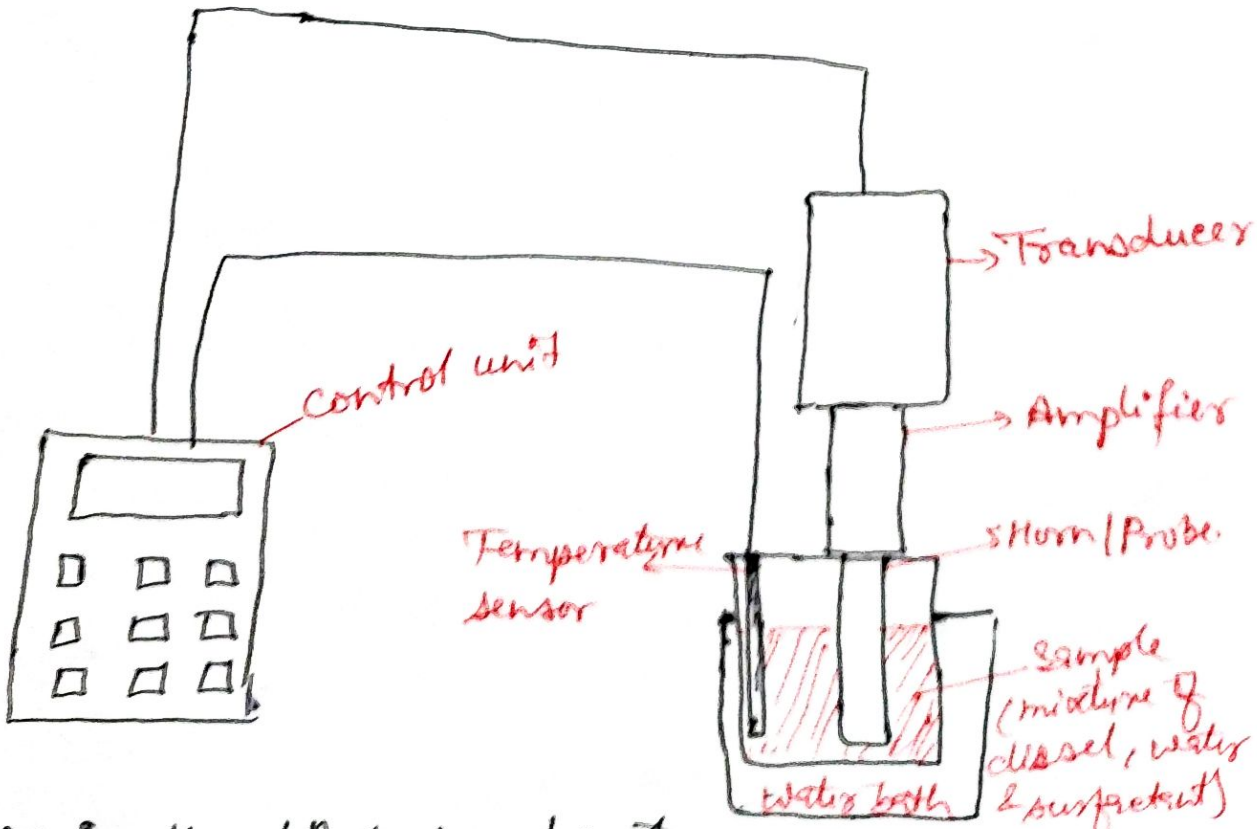
⇒ For extraction applications, formation and collapse of cavitation bubbles depends upon.

⇛ Ultrasonic characteristic. (eg- frequency & intensity)

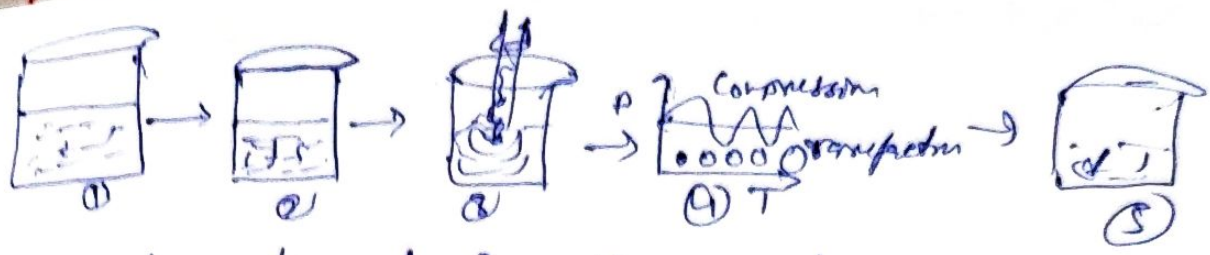
⇛ Solvent / medium properties. for example -

- Vapour pressure governs intensity of collapse
- Surface tension and viscosity govern the transient threshold of cavitation

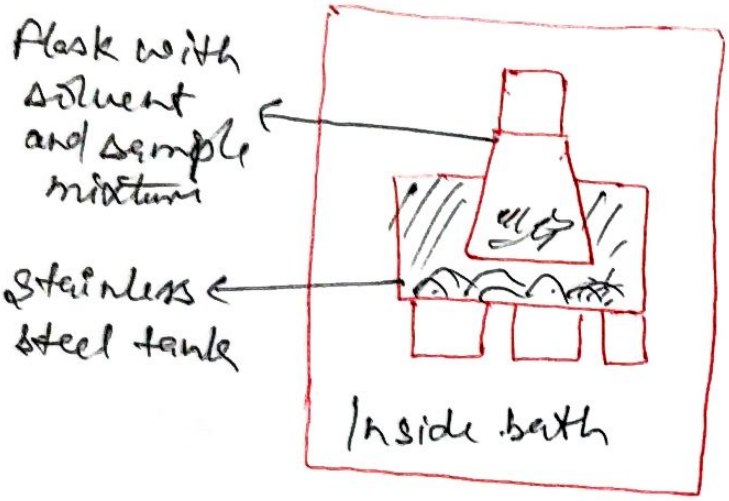
⇛ Ambient condition s. (eg- temperature & pressure).

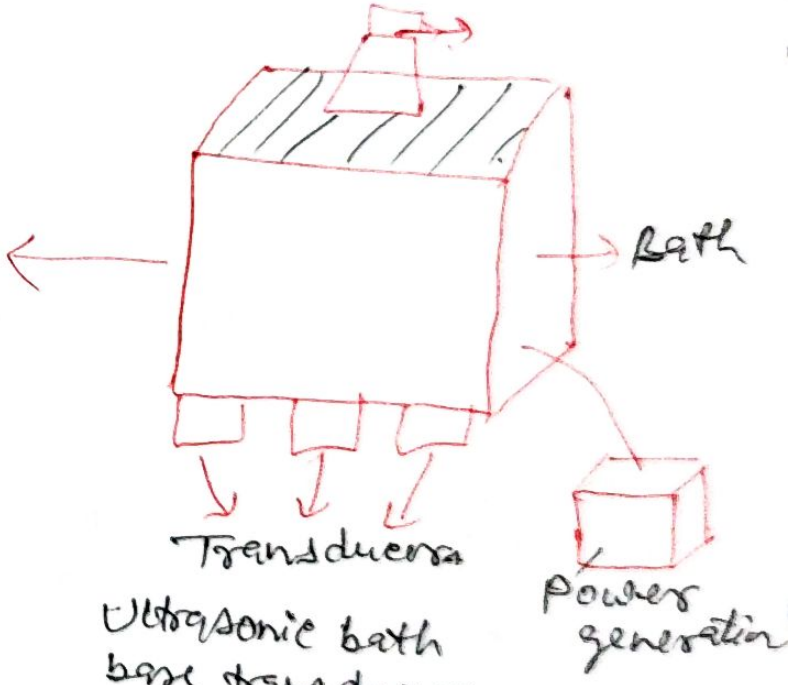


Ultrasonic Horn / Probe based system



(1) Sample placed in the vessel.
(2) Extraction solvent is added.
(3) Sonotrode is placed in contact with sample. sonication of
(4) Ultrasound cavities propogation and implosion lead to Sample localized high temperature and pressure resulting enhanced extraction
(5) Sample ready for clean up process.





Ultrasonic bath

Advantages of UAE :-

(1) It is cheap and has low Instrumental require.
(2) UAE significantly ↓ extraction time & ↑ extraction yield
(3) UAE does not use any heat
(4) Safer solvents, reduce the quantity of solvent used.
(5) Improve the extraction effeciency of solvent.

* Disadvantage = This method is not useful for Industrial purpose

* Application of UAE ⇒

① Ultrasound as a laboratory-based technique for assisting extraction from plant material.

② In analytical chemistry for sample preparation is common in pharmaceutical, Nutraceuticals, cosmetic and other chemical industries.

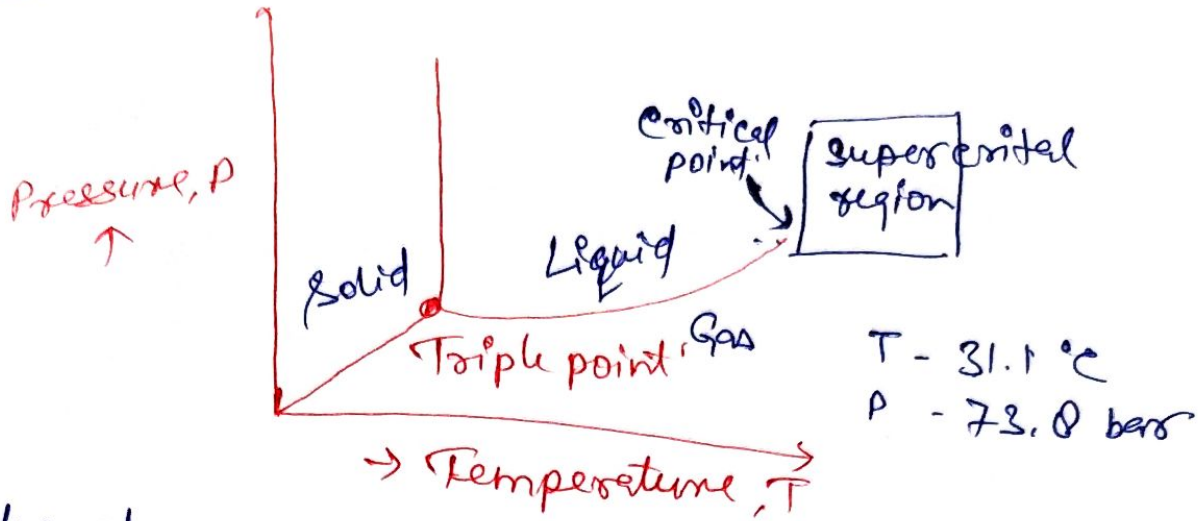
③ Applied as a pretreatment for extraction of essential oils and lipophilic compounds.

④ Improved extraction of total fat from oleaginous seeds. (sunflower, rape & soyabean).

⑤ Application of Ultrasound along with enzymes is reported to enhance extraction yields of polysaccharides.

⇒ Supercritical fluid Extraction :- (SFE) detailed pathak

- The SFE is a comparatively recent method of extraction of crude drugs.
- Certain gases behave like a free flowing liquids or supercritical fluids at the critical point of temperature and pressure. Such supercritical fluid have a very high penetration powers & extraction efficiency.
- The gases lik CO_2 are held as a supercritical fluid at the critical point of 73.83 bar pressure and 31.06 °C temperature.
- At this critical point CO_2 behaves as a liquified gas or free-flowing liquid and assists the extraction of the phytochemical constituents from the crude drugs.
- The phase diagram of CO_2 indicates the characteristic areas for deodorization, extraction & fractionation



- The advantages of CO_2 in supercritical fluid extraction are that it is sterile and bacteriostatic.
- It is noncombustible and non explosive. CO_2 harmless to environment and no waste products are generated during the process, and it is available in large amount under favorable condition.
- The mixture to be fractionated is passed in the extraction column along the length of which the heater is located.
- The operating conditions, i.e. pressure and temperature are selected. In the pressure controlled type of extract the solⁿ is just expanded in the seperation stage to precipitate the extract and then again the gas is recompressed for recycle.
- In temperature control type operation, the extract is precipitated by heating the solution which lower the solvent density.
- The density is then increased by isobaric cooling for recycling operation of supercritical fluid extraction system controlled by PC.

- PC is used to set the operating condition like pressure, temperature and flow rate.
- PC is programmed to safely shut down the unit in case of overpressure or over temperature situations.

Applications of SFE :-

- ⇒ Used in the decaffeination of coffee, extraction of pyrethrins and for the production of terpeneless oil
- ⇒ Removing of odours or still notes from the freshly distilled essential oil.
- ⇒ Selectively extracting larger proportions of active ingredients than conventional methods of hydro-distillation or extraction i.e.- in case of acorone from acorus calamus, matricin and bisabolol from chamomile flowers, heat labile sesquiterpene hydrocarbons of valerian and Nardostachys species.
- ⇒ Extraction of Taxol (Diterpene), primrose (feed oil)

Advantages of SFE :-

- ⇒ Higher diffusion rates than liquid solvents.
- ⇒ Lower viscosities than liquid solvents.
- ⇒ Higher vapour pressure than liquid solvents.
- ⇒ Higher densities compared to gases, higher solvation power
- ⇒ Solubility & selectivity can be controlled by modifier of parameters.
- ⇒ Low polarity of CO_2 can be modified with co-solvents.
- ⇒ Suitable for heat sensitive compounds.

Disadvantages of SFE :-

- CO_2 which is the most commonly used solvent has low polarity and hence cannot extract polar compounds
- Presence of H_2O may cause problems Unpredictability of matrix effect
- Need for specialized / expensive equipment

* Types of Extracts :-

* Aquous extracts :- These are the extracts which are medicinal preparations Intended to be used immediately after prepgration or to be preserved for use.

* Decoction :- This is the ancient and more popular process of extracting water soluble and heat stable constituents from crude drugs by boiling in water for about 15 minutes.

- The boiled crude drugs water mixture is then cooled filtred and sufficient volume of cold water is passed through the drug to produce the required volume.

* Infusion :- An Infusion is generally a dilute solution of the readily soluble constituent of crude drugs. It is nothing but a type of periodic maceration of the drug with either cold or boiling H_2O.

- The Infusion is filtered to remove the crude vegetable material and then produced in a required volume by addition of H_2O

Digestion :- Digestion is also type of maceration in which moderate heating is preferred during extraction. Heating causes the digestion of drug material and increases the solvent efficiency
- It is preferred for the drugs in which the use of moderately elevated temperature does not cause the degradation of constituents

⑤ Tinctures :- Tinctures are the alcoholic or hydro-alcoholic solutions prepared from crude drugs or from the pure organic or inorganic substance
- Tincture of crude drugs may contain 10-20 g of drug per 100 ml of tincture.
- The methods used for the preparation of tinctures are ① maceration and ② percolation
- Iodine tincture is an example of inorganic pharmaceuticals, belladona tincture is prepared by percolation while compound benzoin tincture, sweet orange peel tincture are prepared by maceration.

⑥ Liquid extracts :- The liquid extracts are also termed as fluid extracts in some official books like U.S.P. It is a liquid preparation of crude drugs which contain ethyl alcohol as a solvent and preservative.
- It may contain active constituents to the extent of 1gm of drug per ml. Pharmacopoeial liquid extracts are prepared by the percolation or modified percolation technique

Soft extract :- The extracts which are produced as semisolid or liquids of syrupy consistency are termed as soft extracts. Used in variety of dosage forms ranging from ointment suppositories. or can be used in the preperation of some other pharmaceuticals. Ex- Glycyrrhiza extract USP comes in the form of soft extract.

* Dry extracts — Dry extracts are also known as the powdered extracts or dry powders.

- The total extracts obtained by using suitable process of extraction, are filtered, concentrated preferably under vacuum and dried completely.
- The tray drying or spray drying is used for making dry extracts
- Powdered extracts are used in a solid, dry dosage forms like capsules, powders or tablet.
- The belladona extract, Hyoscymus extract are the official dry extracts.