

## Advance Techniques of Extraction of Phyto Chemicals

Prof. Masarrat Mukadam<sup>1\*</sup>, Mr. Ansari Shaad<sup>2</sup>, Mr. Ansari Fuzail<sup>3</sup>, Ms. Khan Sabiha<sup>4</sup>

Anjuman-I-Islam's Kalsekar Technical Campus, School of Pharmacy, New Panvel, Khandagaon near Thana Naka District Raigad, Maharashtra, India.

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### ABSTRACT

In recent years, variety of extraction techniques has been introduced for the recovery of organic compounds. Extraction Methods are widely used in various Industries for Separation of components and has wide range of applications. Details of basic theories applicable to types of Extraction such as - Liquid- Liquid Extraction, Solid Phase Extraction, Solid Liquid Extraction and Supercritical Extraction, etc. including the choice of solvent, procedure, respective advantages disadvantages and their applications are explained. Finally, the specific extraction techniques such as Microwave Extraction, Ultrasonic Extraction, Pressurized Fluid Extraction and Soxhlet Extraction along with their applications are also explained.

**KEY WORDS** – extraction, methods, advantages, applications

### I. INTRODUCTION

In Extraction the mixture of substances is dissociated, by dissolving each component with one or other solvents which yields two phases – Raffinate Phase (rich in Feed Solvent) and Extract Phase (rich in Solute). When the Relative Volatility is 1 the separation of the components in the mixture is not possible by Distillation and when relative Volatility is Greater than 1 Extraction method is used for separation of the components. Also, when the Distillation Method used is too expensive, Extraction process is opted.

### † CLASSIFICATION OF EXTRACTION TECHNIQUES

#### Classification of Extraction techniques



### † FACTORS AFFECTING EXTRACTION PROCESS

Factors affecting selection of an extraction process

- CHARACTER OF HERB:-** The knowledge of pharmacognosy of the herb is essential to select the right method of extraction process. The maceration process is used when the herb is soft, unorganized un powderable and to

avoid powdering of it. The percolation process is used when the herb is hard and tough.

- THERAPEUTIC VALUE OF THE HERB:-** When the herb has considerable therapeutic value, the maximum extraction is required, so the percolation process is used e.g. Belladonna. In case the herb has little therapeutic value, the efficiency of extraction is unimportant, and

maceration process can be used to extract the herb.

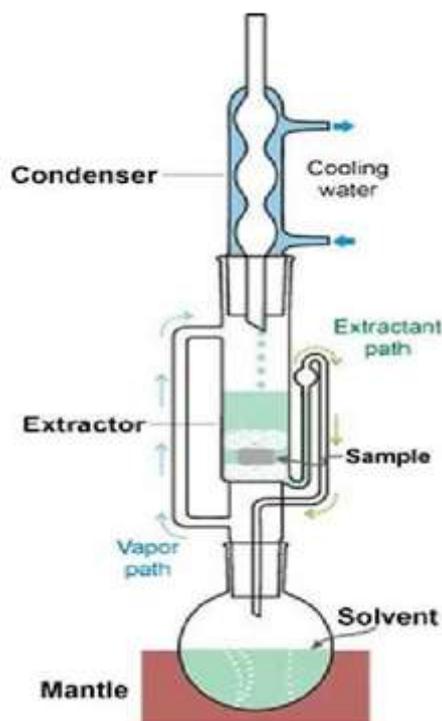
3. **STABILITY OF HERB:-** Continuous hot extraction process should be avoided when the constituents of the herb are thermo labile in nature; in that case maceration or percolation process may be used to extract the active constituents of the herb.
4. **SOLVENT:-** If water is used as a solvent the maceration process should be recommended. The percolation process should be preferred if non-aqueous solvents are used for extraction.
5. **CONCENTRATION OF PRODUCT:-** The dilute products such as tinctures can be made by using maceration or percolation process, depending on the other factors. For semi concentrated preparations, such as concentrated infusions, double or triple maceration process can be used. The liquid extracts or dry extracts which are concentrated

preparations are prepared by using percolation process.

#### ❖TYPES OF EXTRACTION PROCESS

1. **Soxhlet extraction** is only required where the desired compound has a limited solubility in a solvent, and the impurity is insoluble in that solvent. If the desired compound has a high solubility in a solvent then a simple filtration can be used to separate the compound from the insoluble substance. The advantage of this system is that instead of many portions of warm solvent being passed through the sample, just one batch of solvent is recycled.

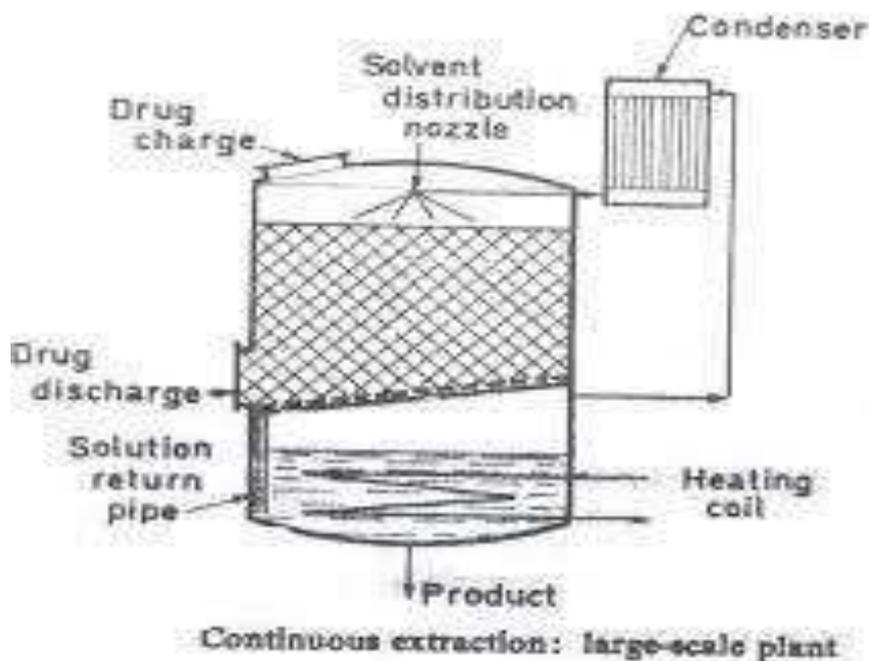
2. **Maceration** In (for fluid extract), whole or coarsely powdered plant-drug is kept in contact with the solvent in a stoppered container for a defined period with frequent agitation until soluble matter is dissolved. This method is best suitable for use in case of the thermolabile drugs.





**3. Decoction** This method is used for the extraction of the water soluble and heat stable constituents from crude drug by boiling it in

water for 15 minutes, cooling, straining and passing sufficient cold water through the drug to produce the required volume



**4. Infusion:** It is a dilute solution of the readily soluble components of the crude drugs. Fresh infusions are prepared by macerating the solids for a short period of time with either cold or boiling water.

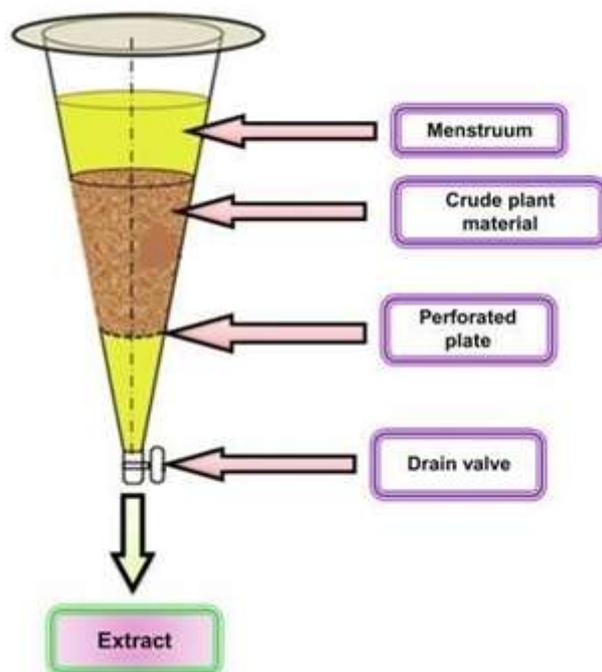


**5. Digestion:** This is a kind of maceration in which gentle heat is applied during the maceration extraction process. It is used when moderately temperature is not objectionable and the solvent efficiency of the menstrum is increased thereby.



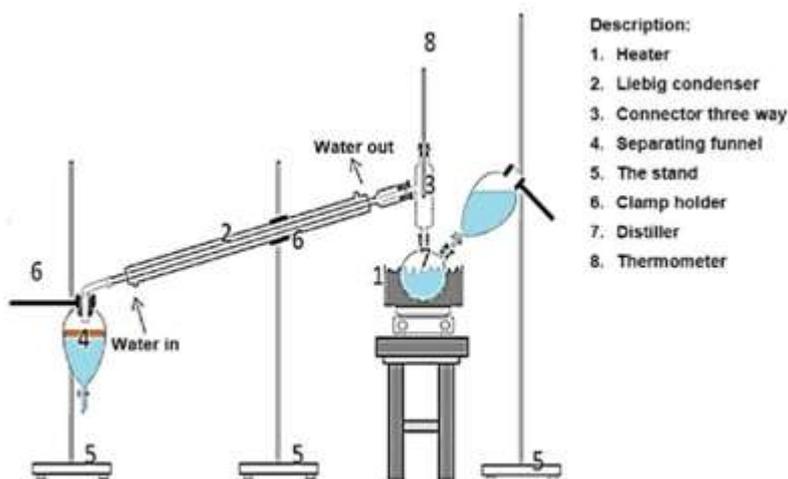
- 6. Sonication:** The procedure involves the use of ultrasound with frequencies ranging from 20 kHz to 2000 kHz; this increases the permeability of cell walls and produces cavitation. Although the process is useful in some cases, like extraction of rauwolfia root, its large-scale application is limited due to the higher costs. One disadvantage of the procedure is the occasional but known deleterious effect of ultrasound energy (more than 20 kHz) on the active constituents of medicinal plants through formation of free radicals and consequently undesirable changes in the drug molecules.
- 7. Percolation:** This is the procedure used most frequently to extract active ingredients in the preparation of tinctures and fluid extracts. A percolator (a narrow, cone-shaped vessel open at both ends) is generally used. The solid

ingredients are moistened with an appropriate amount of the specified menstrum and allowed to stand for approximately 4 h in a well closed container, after which the mass is packed and the top of the percolator is closed. Additional menstrum is added to form a shallow layer above the mass, and the mixture is allowed to macerate in the closed percolator for 24 h. The outlet of the percolator then is opened and the liquid contained therein is allowed to drip slowly. Additional menstrum is added as required, until the percolate measures about threequarters of the required volume of the finished product. The marc is then pressed and the expressed liquid is added to the percolate. Sufficient menstrum is added to produce the required volume, and the mixed liquid is clarified by filtration or by standing followed by decanting.



**8. Hydro distillation** is a traditional method for extraction of plants materials that doesn't used organic solvents. In hydro distillation, plant

materials are packed in a still compartment and water is added in sufficient amount, and then brought to boil.

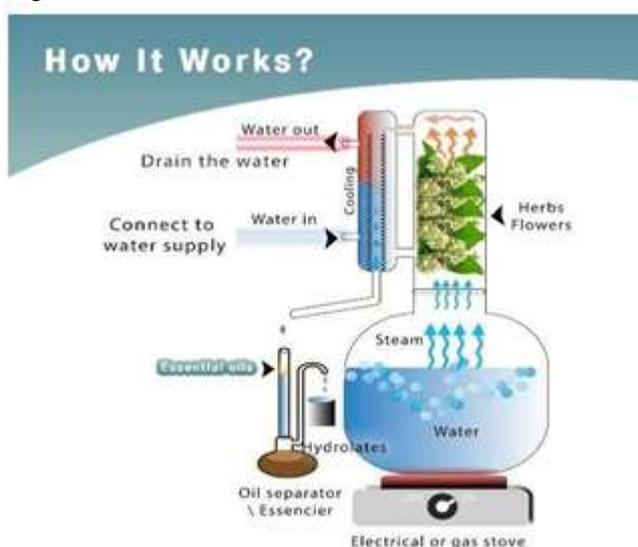


Alternatively, direct steam is injected into the plant sample. Hot water and steam act as the main influential factors to free bioactive compounds of plant tissue. Indirect cooling by water condenses the vapor mixture of water and oil. Hydro Distillation is potentially a very useful method to extract essential oil from various plants and from their different parts. The yield is

dependent on various parameters like weight of raw material, volume of water, size of raw material and nature of raw material [15]. Hydro distillation involves three main physio chemical processes; Hydro diffusion, hydrolysis and decomposition by heat. At a high extraction temperature some volatile components may be lost.

**9. Direct steam distillation** As the name suggests, direct steam distillation is the process of distilling plant material with steam generated outside the still in a steam generator generally referred to as a boiler. As in water and steam distillation, the plant material is supported on a perforated grid above the steam inlet. A real advantage of satellite steam

generation is that the amount of steam can be readily controlled. Because steam is generated in a satellite boiler, the plant material is heated no higher than 100° C and, consequently, it should not undergo thermal degradation. Steam distillation is the most widely accepted process for the production of essential oils on large scale.



Throughout the flavor and fragrance supply business, it is a standard practice.

**10. Tinctures:** Tinctures are the alcoholic or hydroalcoholic solutions prepared from crude drugs or from the pure organic or inorganic substances. Tinctures of crude drugs may contain 10–20 g of drug per 100 ml of tincture.

The methods used for the preparation of tinctures are: maceration and percolation. Iodine tincture is an example of inorganic pharmaceuticals, belladonna

tincture is prepared by percolation while compound benzoin tincture, sweet orange peel tincture are prepared by maceration.

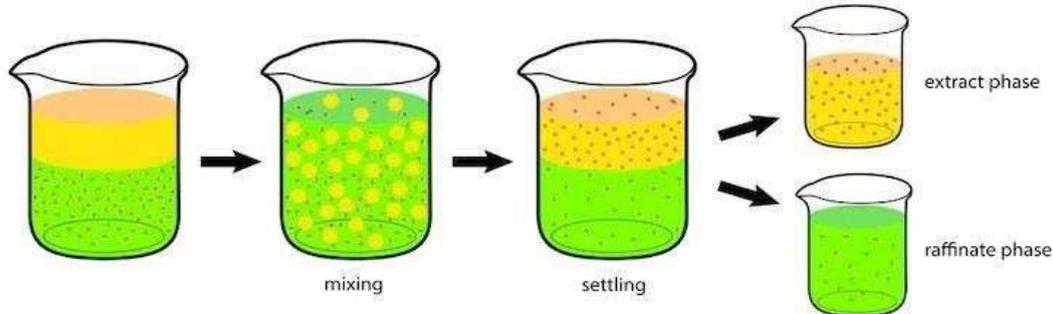


**11. Liquid Extracts:** The liquid extracts are also termed as fluid extracts in some official books

like USP. 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 1 g of drug per ml. Pharmacopeial

liquid extracts are prepared by the percolation or modified percolation techniques.



**12. Soft Extract:** The extracts which are produced as semisolid or liquids of syrupy consistency are termed as soft extracts. These extracts are

used in the variety of dosage forms ranging from ointments, suppositories or can be used in the preparation of some other pharmaceuticals



**13. Dry Extract:** 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. Just like soft

extracts, these powdered extracts can be used for the manufacture of some medicinal preparations. Powdered extracts are preferably used into a solid, dry dosage forms like capsules, powders or tablets. The Belladonna extract, Hyoscyamus extract are the official dry extracts.



#### 14. ULTRASONIC-ASSISTED EXTRACTION

Ultrasonic-assisted extraction is an effective and rapid technique for extracting cannabis concentrates. It does improve the diffusion process by accelerating mass transfer within the plant materials, causing the cell walls to rupture and to release the desired compounds. A vibrating ultrasonic probe immersed in a liquid will transmit alternating high and low pressure waves.

These fluctuations cause the liquid molecular cohesive forces to break- down, pulling apart the liquid and creating millions of micro-bubbles (cavities), which expand during the low pressure phases and implode violently during the high pressure phases. As the bubbles collapse, millions of microscopic shock waves, micro jet streams, and eddies are generated at the implosion sites and propagated to the surrounding medium. Although

this phenomenon, known as cavitation, lasts but a few microseconds, and the amount of energy released by each individual bubble is minimal, the cumulative amount of energy generated by the imploding cavities is extremely high promoting surface peeling, erosion, and particle breakdown. By disrupting the cell in this manner, solvent penetration is enhanced, accelerating the release of bioactive compounds and other components from the biological matrix into the extraction medium. Because focused ultrasound extraction provides more than 100 times the radiated energy generated in an ultrasonic bath, it is ideally suited for the

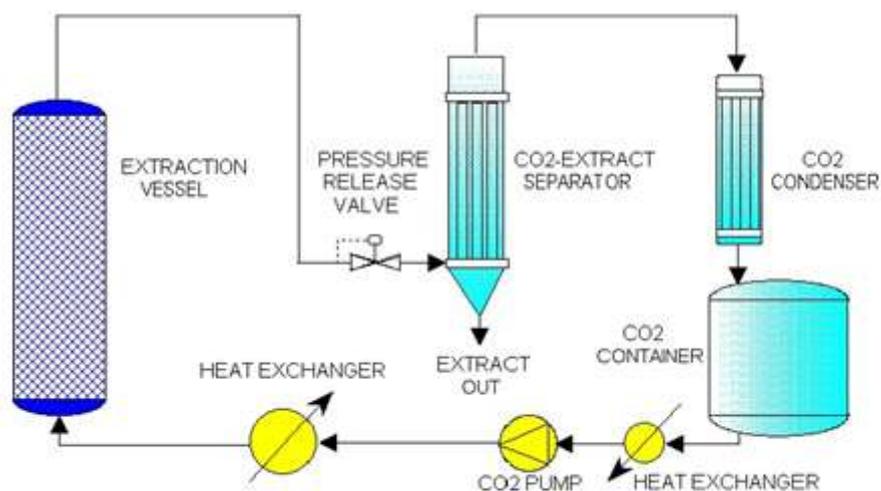
extraction of beneficial cannabinoid. When extracted properly, the resulting concentrate is reminiscent of the cannabis strain it was extracted from – the taste smell and effects are simply amplified due to a larger concentration by weight. On average, cannabis plant buds will yield about 15% of extracted concentrate. As expected, the quality of the extract and bioavailability will greatly depend on the amount and potency of the plant matter/trichrome (the crystalline hair-like structures coating the outside surface of the flowers) used.



### 15. SUPERCRITICAL FLUID EXTRACTION

Supercritical fluid extraction is the process of separating one component (the extractant) from another (the matrix) using supercritical fluids as the extracting solvent. Extraction is usually from a solid matrix, but can also be from liquids. SFE can be used as a sample preparation step for analytical purposes, or on a larger scale to either strip unwanted material from a product (e.g. decaffeination) or collect a desired product (e.g.

essential oils). These essential oils can include limonene and other straight solvents. Carbon dioxide (CO<sub>2</sub>) is the most used supercritical fluid, sometimes modified by co-solvents such as ethanol or methanol. Extraction conditions for supercritical carbon dioxide are above the critical temperature of 31 °C and critical pressure of 74 bar. Addition of modifiers may slightly alter this. The discussion below will mainly refer to extraction with CO<sub>2</sub>, except where specified.



#### ❖ADVANTAGES OF EXTRACTION PROCESS

The advantages of extraction are low working temperatures, the feasibility of obtaining substances from dilute solutions, the possibility of separating mixtures consisting of components with similar boiling points and azeotropic mixtures, the possibility of combining extraction with other technological processes, such as rectification and crystallization, the simplicity of the equipment used, and the ease of automating the various steps. A shortcoming in many cases is the difficulty in completely removing the extractant from the extracted substances.

#### ❖DISADVANTAGES OF EXTRACTION

1. The samples are heated to a high temperature for a relatively long period thus the risk of thermal destruction of some compounds cannot be overlooked if the plant material contains heat labile compounds. 2. The extraction time is lengthy and the process is labor intensive 3. The process allows manipulations of limited variables. The time and the requirement of a large amount of solvent result in wide criticism of Soxhlet extraction technique

#### ❖APPLICATIONS OF EXTRACTION

##### Extracting Natural Compounds

Fruit and plant leaves are primarily composed of cellulose and water, but also contain "essential oils," a greasy mixture of compounds that capture the "essence" of the plant material's smell and taste. Orange oil is roughly 95% limonene (Figure 4.3b), and due to its nonpolar structure, can be extracted from its rind into an organic solvent like hexanes or dichloromethane. The oil can then be

concentrated and used to flavour or scent foods, cleaning supplies, and candles.

##### Transferring Compounds From Layers

Another method for extracting essential oils from fragrant plant materials is through steam distillation (Figure 4.4b). This process often results in the lovely smelling compounds suspended in the aqueous distillate (Figure 4.4c). In order to concentrate the oil, the aqueous suspension is often extracted with a low-boiling organic solvent (Figure 4.4d), which can then be easily removed from the oil.

##### Selective Removal of Components

When conducting an experiment that synthesizes a chemical product, a reaction is often complete whenever stirring or heating is ceased. And yet, there are always more steps in the procedure! What commonly happens directly afterwards is to "**work-up**" the reaction in some way. A work-up refers to methods aimed at isolating the product from the reaction mixture, and often begins by using a separatory funnel and extractions.

## II. CONCLUSION

In the extraction process, a solute is transferred from one phase to another to separate it from unreacted starting materials or impurities. Extraction is also used to facilitate the isolation of a solute from a reaction solvent that is difficult to remove by evaporation, such as a solvent with a high boiling point. Two phases result from the extraction step: one enriched (extract Phase) and the other depleted (RAFFINATE Phase) in the components to be separated, respectively.



Afterwards in order to regenerate the solvent, another separation step (e.g. distillation) is finally required.

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