Extraction; maceration and percolation

By
Dr. Mohammed Sattar
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Outlines

- Extraction
- Method selection
- Maceration
- Percolation
Extraction Methods for Preparing Solutions

- The sole purpose of such basic extraction procedures for crude drugs are to obtain the therapeutically desirable portion and eliminate the inert material by treatment with a selective solvent known as the Menstruum.

- Certain pharmaceutical preparations are prepared by extraction, that is, by withdrawal of desired constituents from crude drugs through the use of selected solvents in which the desired constituents are soluble.

- Types of extraction procedures play a decisive role for the qualitative and quantitative composition of the extracts.

- The standardized extracts, thus obtained are further processed for inclusion in other dosage forms such as Tablets and Capsules containing several groups of plant’s metabolites.

- Because each crude drug contains a number of constituents that may be soluble in a given solvent, the products of extraction, termed extractives, do not contain just a single constituent but rather varying constituents, depending on the drug used and the conditions of the extraction.

- Tinctures, fluidextracts, and extracts are the pharmaceutical products most commonly prepared from extractives.

- The solvent systems used in extraction are selected on the basis of their capacity to dissolve the maximum amount of desired active constituents and the minimum amount of undesired constituents.
In drug extraction, the solvent or solvent mixture is referred to as the **menstruum**, and the plant residue, which is exhausted of active constituents, is termed the **marc**.

The selection of the menstruum to use in the extraction of a crude drug is based primarily on its ability to dissolve the active constituents.

Although water and alcohol and to a lesser extent glycerin are probably the most frequently employed solvents in drug extraction, acetic acid and organic solvents like ether may be used for special purposes.

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**Methods of Extraction**

(1) Maceration  
(2) Percolation  
(3) Decoction  
(4) Digestion  
(5) Infusion
Methods of Extraction

• The principal methods of drug extraction are maceration and percolation.

• Factors affecting method selection:
  1. The nature of the crude drug,
  2. The drug adaptability to each of the various extraction methods, and
  3. The interest in obtaining complete or nearly complete extraction of the drug.

Character of Drug

➢ If hard and tough (such as nux vomica) use percolation.
➢ If soft and parenchymatous (such as gentian) use maceration.
➢ If ‘unpowderable’ (such as squill) use maceration.
➢ If an ‘unorganized drug (such as benzoin) use maceration.
➢ If preferable to avoid powdering (such as senna fruits) use maceration.

Thus, knowledge of the pharmacognosy of the drug is essential to selection of the extraction process that will give the best results.
Therapeutic value of the drug

• When the drug has considerable therapeutic value, the maximum extraction is required, so that percolation is used, as in belladonna.

• If the drug has little therapeutic value, however, the efficiency of extraction is unimportant and maceration is adequate; for example, “flavours” (lemon), or “bitters”, (gentian).

Stability of drug

• Continuous extraction should be avoided when the constituents of the drug are thermo-labile.

Solvent

• If the desired constituents demand a solvent other than a pure boiling solvent or an azeotrope, continuous extraction should be used.

• Recovery of solvent from the marc

• The residue of the drug after extraction (often known as the marc) is saturated with solvent and if economic the latter is recovered.
Cost of drug

- From the economic point of view, it is desirable to obtain complete extraction of an expensive drug, so that percolation should be used; Ginger is an example.

- For cheap drugs, the reduced efficiency of maceration is acceptable in view of the lower cost of the process. In particular, the cost of size reduction to a powdered state is avoided, whereas this is a significant part of the percolation process.

Concentration of product

- **Dilute products** such as tincture can be made by maceration or percolation, depending on the previous factors.

- For **semi-concentrated** preparations (concentrated infusions, for e.g.) the more efficient percolation process is used) unless the drug cannot be powdered or is not worth powdering, when double or triple maceration is chosen.

- **Concentrated preparations**, of which liquid extracts or dry extracts are example, are made exclusively by percolation, with the exception that continuous extraction can be used if the solvent is suitable and the constituents are thermo-stable.
(1) Maceration: Steady state extraction

• Term derived from Latin word “macerare” meaning to soak.

• It is a process in which the properly comminuted drug is permitted to soak in the menstruum until the cellular structure is softened and penetrated by the menstruum and the soluble constituents are dissolved.

• For drugs containing little or no cellular material, such as benzoin, aloe, and tolu, which dissolve almost completely in the menstruum, maceration is the most efficient method of extraction.

• Maceration is usually conducted at a temperature of 15°C to 20°C for 3 days or until the soluble matter is dissolved.

• Examples:
  1. Compound Benzoin,
  2. Sweet orange Peel Tincture;
  3. Compound Cardamon;
  4. Tolu Balsam Tincture
Plant Material: cut into small pieces
Placed in a closed vessels with menstruum
Standing with occasional shaking
Liquid strained off and clarified
Evaporation and concentration

Modifications of the General Processes of Maceration

- **Repeated maceration** may be more efficient than a **single maceration**, since an appreciable amount of active principle may be left behind in the first pressing of the marc.
- The repeated maceration is more efficient in cases where active constituents are more valuable.
- **Double maceration** is used for concentrated infusions which contain volatile oil, e.g. Concentrated Compound Gentian Infusion.
- Where the marc cannot be pressed, a process of **triple maceration** is sometimes employed.
- The total volume of solvent used is however large and the second and third macerates are usually mixed and evaporated before adding to the first macerates.
• This precludes, the use of the process for preparations containing volatile ingredients.

• In a few cases, it is desirable to change the physico-chemical nature of the solvent during a single maceration process.

• **Opium Tincture** is prepared by using change of the physico-chemical nature of the solvent as indicated below:

  - Sliced opium disintegrated in boiling water and left to cold for 6h
  - 90% Alcohol is added to the cold mix and macerate for 24h
  - The addition of alcohol prevent the formation of gummy material in the final product.

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**Circulatory extraction**

• The efficiency of extraction in a maceration process can be improved by arranging for the solvent to be continuously circulated through the drug.

• Solvent is pumped from the bottom of the vessel to the inlet where it is distributed through spray nozzles over the surface of the drug.

• The movement of the solvent reduces boundary layers, and the uniform distribution minimizes local concentration in a shorter time.
Multiple stage extraction

- Like the normal maceration process,

- however, extraction is incomplete, since mass transfer will cease when equilibrium is set up. This problem can be overcome by using a multistage process.

- The equipment needed for this method is a circulatory extractor connected to number of tanks to receive the extracted solution.
Procedures

1. Fill extractor with drug, add solvent to circulate. Run off to receiver 1.
2. Refill extractor with solvent and circulate. Run off to receiver 2.
3. Refill extractor with solvent and circulate. Run off to receiver 3.
4. Remove drug from extractor and recharge. Return solution from 1 to extractor. Remove for evaporation.
5. Return solution from 2 to extractor and circulate. Run off to receiver 1
6. Return solution from 3 to extractor and circulate. Run off to receiver 2
7. Add fresh solvent to extractor and circulate. Run off to receiver 3
8. Remove drug from extractor and recharge. Repeat cycle.

Advantages

1. The drug is extracted as many times as there are several receivers.
2. The last treatment of the drug before it is discharged is with fresh solvent, giving maximum extraction.
3. The solution is in contact with fresh drug before removal for evaporation, giving the highest possible concentration.
**(2) Percolation: Exhaustive extraction**

- The term percolation, from the Latin *per*, meaning *through*, and *colare*, meaning *to strain*.

- It may be described generally as a process in which a comminuted drug is extracted of its soluble constituents by the slow passage of a suitable solvent through a column of the drug.

- The drug is packed in a special extraction apparatus termed a percolator, with the collected extractive called the percolate.

- Most drug extractions are performed by percolation.

- In the process of percolation the flow of the menstruum over the drug column is generally downward to the exit orifice, drawn by the force of gravity as well as the weight of the column of liquid.

- In certain specialized and more sophisticated percolation apparatus, additional pressure on the column is exerted with positive air pressure at the inlet and suction at the outlet or exit.
Percolators for drug extraction vary greatly as to their shape, capacities, composition, and, most important utility.

Percolators employed in the large-scale industrial preparation of extractive are generally stainless steel or glass lined metal vessels that vary greatly in size and in operation.

Percolation on a small scale generally involves the use of glass percolators of various shapes for extraction of small amounts (perhaps up to 1000 g) of crude drug.

Different shapes of percolator

1. Cylindrical, with little, if any taper except for the lower orifice also called Oldberg percolator.
2. Roundish, but with a definite taper downward.
3. Conical or funnel shape.
   - The cylindrical percolator is particularly suited to the complete extraction of drugs with a minimal expenditure of menstruum.
• **The choice of type of Percolator depends upon**
  1. Nature of the drug
  2. Type of product prepared
  3. Quantity of drug to be extracted

• **Processes and steps of Percolation**
  1. Preparation of the dried crude drug for percolation
     a. Powdering
     b. Moistening
  2. Packing the percolator
  3. Period of Maceration
  4. Percolation and collection of percolate
  5. Adjustment of concentration of percolation as required

**Modifications**

• In general process of percolation the following problems may arise:

a) If the active substances are ** thermo-labile**, evaporation of large volume of dilute percolate, may result in partial loss of the active constituents.

b) In the case of alcohol- water mixture, evaporation results in preferential vaporization of alcohol leaving behind an almost aqueous concentrate ** which may not be able to retain the extracted matter in solution and hence get precipitated. **
Reserved Percolation

- In this case the extraction is done through the general percolation procedure.
- At the last, the evaporation is done under reduced pressure in equipment such that all the water is removed.
- This is then dissolved in the reserved portion which is strongly alcoholic and easily dissolves the evaporated portion with any precipitation.

(3) Decoction
This method involves the placement of plant material in water, then boiling the mixture for about 15 minutes, followed by filtrations to obtain the extractive and removing the marc.

(4) Digestion
Maceration with continued heating during maceration period. The temperature is between 40 °C– 60 °C.

(5) Infusion
Involves first macerating the drug with cold water, followed by the addition of boiling water in an amount equal to 90% of the desired volume.
Thanks for your attention