Pharmaceutical and Cosmetic Preparations

3rd Stage

College of Pharmacy

Lecture: 2- Emulsions (part II)

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Preparation Methods

- Various methods are employed for emulsion preparation, ranging from small-scale laboratory techniques to large-scale industrial processes.
- Each method has its **advantages** and is **chosen** based on *the nature of components and desired emulsion characteristics*

Small-Scale Emulsion Preparation Methods

- **Laboratory Equipment:**
- Small-scale emulsions can be prepared using equipment like:
  1. Dry Wedgwood or porcelain mortar and pestle,
  2. Mechanical blenders such as milkshake mixers, hand homogenizers, or
  3. Simple prescription bottles.
  4. Beaker
- **Laboratory Techniques:**
  - In the small-scale extemporaneous preparation of emulsions using polysaccharide emulsifier (gum), and the oil is liquid at room temperature.
  - Three methods may be used:
    1. **The continental or dry gum method**
    2. **The English or wet gum method**
    3. **The bottle or Forbes bottle method**
    4. **Other method :**
       - **I. Beaker method**
       - **II. In situ soap method (Nascent soap method)**
       - **III. Auxiliary methods:**

The continental or dry gum method:

- **The continental or dry gum method** involves triturating the emulsifying agent (e.g., acacia) with oil before adding water (**addition of external phase to the internal phase**).
- The continental method is also referred to as the **4:2:1 method** because for every **four parts** by volume of **oil**, **two parts of water** and **one part of gum** are added in preparing the initial or primary emulsion.
    - For instance, if 40 mL of oil is to be emulsified, 20 mL of water and 10 g of gum would be employed in the primary emulsion.
  - **In this method**, *the acacia or other o/w emulsifier is triturated with the oil* in a perfectly dry **Wedgwood or porcelain mortar** until thoroughly mixed.
  - After the oil and gum have been mixed, the **two parts of water** are added **all at once**, and the mixture is triturated immediately, rapidly, and continuously until the primary emulsion is formed.
    - A mortar with a rough rather than smooth inner surface must be used to ensure proper grinding action and reduction of the globule size.
    - A glass mortar is too smooth to produce the proper reduction of the internal phase.
  - The emulsion is **transferred** to a **graduate** and made to volume with water previously swirled about in the mortar to remove the last portion of emulsion.

- **English or Wet Gum Method:**
  - The English or wet gum method creates a mucilage of the emulsifying agent with water **before** slowly incorporating oil (**addition of internal phase to the external phase**).
    - By this method, the same proportions of oil, water, and gum are used as in the continental or dry gum method, but the *order of mixing is different*.
    - A mucilage of the gum is prepared by *triturating in a mortar granular acacia with twice its weight of water. The oil is then added slowly in portions, and the mixture is triturated to emulsify the oil.*
    - After **all** of the oil has been added, the mixture is thoroughly mixed for several minutes to ensure uniformity.
    - Then, the **other** formulative materials are added, and the emulsion is transferred to a **graduate** and **brought** to volume with water.

Bottle or Forbes bottle method:

- The bottle or Forbes bottle method is suitable for volatile oils or oleaginous substances of low viscosities, involving shaking the mixture in a capped bottle.
  - Powdered **acacia** is placed in a **dry bottle**, two parts of oil are added, and the mixture is thoroughly shaken in the capped container.
  - A volume of water approximately equal to that of the oil is then added in portions and the mixture thoroughly shaken after each addition.
  - When all of the water has been added, the primary emulsion thus **formed** may be diluted to the proper volume with water or an aqueous solution of other formulative agents

Other method of emulsion preparation

Beaker method

- When synthetic or non-gum emulsifiers (solid particles) are used, the proportions given in the previous methods become meaningless.
  - The most appropriate method for preparing emulsions from **surfactants or other non-gum emulsifiers** is to begin by dividing components into water-soluble and oil-soluble components.
  - All **oil-soluble components** are dissolved in **the oily phase in one beaker** and all **water-soluble components** are dissolved **in the water in a separate beaker**.
  - Oleaginous components are melted, and both phases are heated to approximately 70°C over a water bath.
  - The internal phase is then added to the external phase with stirring until the product reaches room temperature.
  - The mixing of such emulsions can be carried out in a beaker, mortar, or blender, or, in the case of creams and ointments, in the jar in which they will be dispensed.

In Situ Soap Method (Nascent Soap Method)

- In situ" means **formed at the site**.
  In this method, the soap (emulsifying agent) is **formed during preparation**, not added separately.
- **Types of soaps produced**
  - This method develops two types of soaps:
    - **Calcium soaps**
    - **Soft soaps**
- Calcium soaps are **water-in-oil (w/o) emulsions**.
  - They are prepared by mixing:
    - A **vegetable oil** (such as olive oil)
    - **Lime water** (Calcium hydroxide solution, $Ca(OH)_2$)
  - **Chemical Reaction:**
    - Vegetable oils contain **free fatty acids** (for example, oleic acid in olive oil).
    - When oleic acid reacts with calcium hydroxide:
      - Oleic acid + Calcium hydroxide → Calcium oleate (soap)
    - The product **calcium oleate** acts as the **emulsifying agent**.
  - So the emulsifier is formed *inside the system itself* — that is why it is called the **nascent soap method**.
  - Because the oil phase is the external phase, this formulation is i*deal where occlusion and skin softening are desired, such as for itchy, dry skin or sunburned skin.*

In Situ Soap Method (Nascent Soap Method)

Example:

Calamine liniment:

Calamine
Zinc oxide aa 80.0 g
Olive oil
Calcium hydroxide solution aa qs ad 1,000.0 mL

- Olive oil reacts with calcium hydroxide
- Calcium oleate forms in situ
- This stabilizes the water-in-oil emulsion
- Calamine and zinc oxide act as soothing and protective agents

Auxiliary methods:

- Instead of, or in addition to, any of the preceding methods, the pharmacist can usually prepare an excellent emulsion ***using an electric mixer or blender***.
- An emulsion prepared by any method can generally **be increased in quality by passing it through a hand homogenizer.**
- In this apparatus, the pumping action of the handle forces the emulsion through a very small orifice that reduces the globules of the internal phase to about **5 µm** and sometimes less.
- The hand homogenizer is **less efficient** in *reducing the particle size of very thick emulsions.*

Important considerations on emulsion preparation

- Prior to mixing, all the water soluble ingredients are dissolved in the aqueous phase and all the oil-soluble components are dissolved in the oil
- Solid substances such as preservatives, stabilizers, colorants, and any flavoring material are usually dissolved in a suitable volume of water (assuming water is the external phase) and **added** as a solution to the **primary emulsion.**
- If fats waxes or SAA that are solids or semisolid at room temperature are needed, warm the two phases, the aqueous phase temperature is raised 2-3C° above that of oil phase, so that no local crystallization of waxes takes place upon mixing of the two phases
- Any substances that might interfere with the stability of the emulsion or the emulsifying agent are **added** as near **last** as is practical.
- Alcohol has a precipitating action on gums such as acacia, thus **no alcohol** or solution containing alcohol should be **added** directly to the primary emulsion, because the total alcoholic concentration of the mixture would be greater at that point than after other diluents were added
- When all necessary agents have been added, the emulsion is transferred to a **graduate** and made to volume with **water previously swirled about in the mortar** to remove the last portion of emulsion.
- **Forbes bottle method** is not suited for viscous oils because they **cannot be thoroughly agitated in the bottle** when mixed with the emulsifying agent.
- When the intended dispersed phase is a **mixture of fixed oil and volatile oil**, the dry gum method is generally employed

o Large-scale preparation

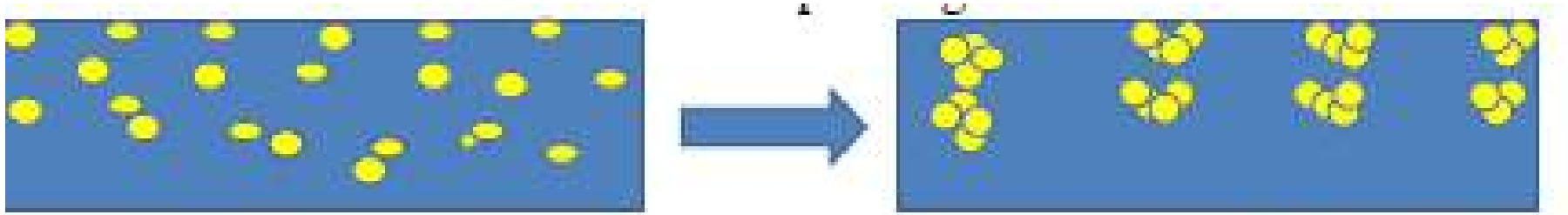
- On a larger scale, **mixing tanks** equipped with **high-speed impellers** are used for emulsion formation. (containing surfactants)
  - Colloid mills or large homogenizers may be employed to refine the emulsion further.
- industrial homogenizers have the capacity to handle as much as 100,000 L of product per hour.

Stability of Emulsions

- The instability of emulsions can be categorized as follows:
  1. Flocculation
  2. Creaming or sedimentation
  3. Coalescence or aggregation
  4. Cracking or breaking
  5. Phase separation

❑ Flocculation

- This process refers to aggregation or joining droplets together (without any change in primary droplet size) to form a large units or clumps (floccules) which rise or settle in the emulsion depending on their densities.



❑ Creaming

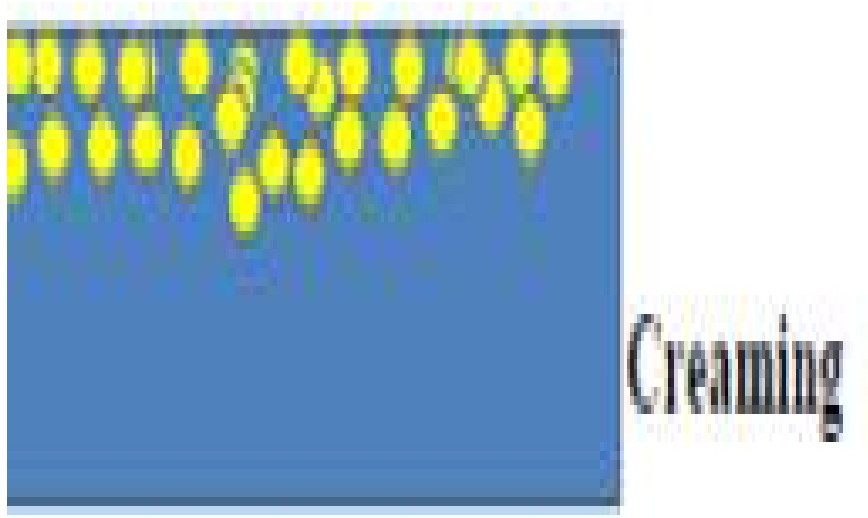
- Aggregates of globules of the internal phase have a greater tendency than do individual particles to rise to the top of the emulsion or fall to the bottom.
- Such a preparation of the globules is termed the creaming of the emulsion, it is a **reversible** process.
- Creamed emulsion can be restored to its original state by gentle agitation, if insufficient shaking is employed before each dose, improper dosage of the internal phase substance may result.
- According to the Stokes equation the rate of separation of the dispersed phase of an emulsion may be related to such factors:
  1. The particle size of the dispersed phase,
  2. The difference in density between the phases,
  3. The viscosity of the external phase.

❑ Creaming

- Larger particle size, greater density difference, and lower viscosity **increase** the rate of creaming.
- Upward creaming takes place in unstable emulsions of the o/w or the w/o type in which the internal phase has a lesser density than the external phase. Downward creaming takes place in unstable emulsions in which the opposite is true

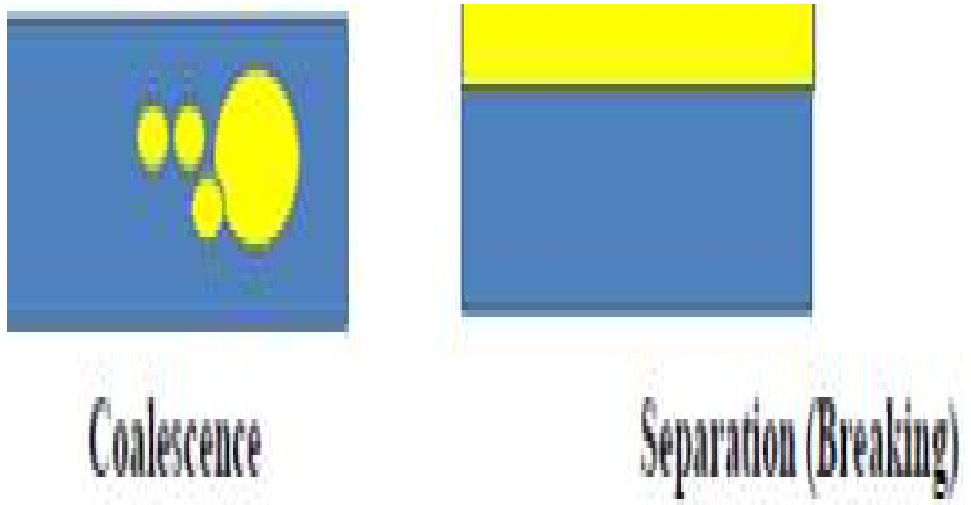
Strategies for enhanced the stability:

1. The globule or particle size should be reduced as fine as is practically possible
2. The density difference between the internal and external phases should be minimal
3. The viscosity of the external phase should be reasonably high.



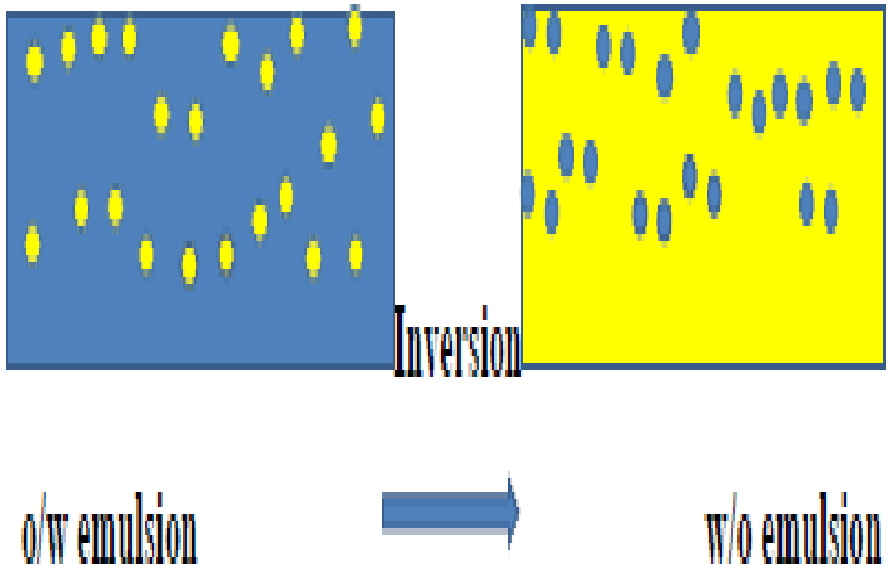
❑ Coalescence and Breaking (Cracking)

- If the droplet contacts another droplet, coalescence will occur to produce a single droplet of greater diameter and, the surface area of the new droplet will be less than the surface areas of the two individual droplets.
- This process will continue until there is complete phase separation (i.e. two liquid layers occur).
- Separation of the internal phase from the emulsion is called **breaking**, and the emulsion is described as being **cracked or broken**.
- This is **irreversible**, because the **protective sheath** about the globules of the internal phase **no longer exists**.
- If an emulsion has cracked it **cannot** be recovered or redispersed by **shaking**



❑ Phase Inversion

- It is the process of an **exchange** between the **disperse phase and the medium**.
- Over time or when conditions change (like composition, temperature, or agitation), an **o/w emulsion can turn into a w/o emulsion**, or vice versa. This switching is called **phase inversion**
- One major reason is **volume fraction**:
  - ➢ When the dispersed phase becomes too large—**more than about 74% of the total volume**—the system becomes unstable.
  - ➢ At this point, it's energetically easier for the phases to flip, so the dispersed phase becomes the continuous phase.



Consideration for temperature, light, environmental factors and storage

- care must be taken to protect emulsions against extremes of cold and heat.
    - **Freezing and thawing** coarsen an emulsion and sometimes break it.
    - Excessive **heat** has the **same** effect.
- The presence of light, air, and contaminating microorganisms can adversely affect the stability of an emulsion; appropriate formulative and packaging steps are usually taken to minimize such hazards to stability.
- For **light-sensitive** emulsions, light-resistant containers are used.
- For emulsions susceptible to **oxidative** decomposition, antioxidants may be included in the formulation and **adequate label warning** provided to ensure that the container is **tightly closed** to air after each use.

Preservation of Emulsions

- Emulsions are **thermodynamically unstable systems** and, in addition to physical instability, they are also **prone to microbial contamination**.
- This is especially true because emulsions often contain **water**, which provides a suitable environment for the growth of microorganisms.
- Many **microorganisms such as molds, yeasts, and bacteria** can grow in emulsions.
- These organisms may **decompose the emulsifying agent**, leading to breakdown of the emulsion and loss of product stability and safety.
- Among these microorganisms, **fungi (molds and yeasts)** are more commonly found in emulsions than bacteria.

➢ Therefore, **fungistatic preservatives** are routinely added.

- A commonly used preservative system is a **combination of methylparaben and propylparaben**, which provides effective protection.
- In **oil-in-water (o/w) emulsions**, these preservatives are usually added to the **aqueous phase**, where microbial growth is most likely to occur.
- For **oral o/w emulsions**, another method of preservation is the addition of **alcohol**. Alcohol in a concentration of about **12% to 15% (based on the volume of the external phase)** is frequently used because it effectively inhibits microbial growth

Therapeutic examples of Oral and Topical Emulsions

- **Oral Emulsions**
  - **Mineral oil emulsion:**
    - Mineral oil emulsion, or liquid petrolatum emulsion, is **an o/w emulsion** prepared from the following materials:
  - Used as a **lubricating cathartic** with enhanced palatability compared to plain mineral oil.
  - Commercial variations may include additional cathartic agents.

Mineral oil	500 mL
Acacia	125 mL
Syrup	100 mL
Vanillin	40 mL
Alcohol	60 mL
Purified water to make	1000mL

Preparation by the dry gum method (4:2:1).

  - **Castor Oil Emulsion:**
    - Indications: Laxative for constipation and colon preparation for diagnostic examinations.
  - **Simethicone Emulsion:**
    - Indications: Defoaming agent for relief of gastrointestinal gas symptoms.

Topical Emulsions

❑ Lotions: Water-based liquid Emulsion

- Many hand and body lotions for dry skin are **o/w emulsions**, which have 70-80% water with fewer oils, with light and fluid texture for easy application and better absorption
- Anti-Itch Moisturizing Lotion with Pra**moxine Hydrochloride** (anesthetic), Relieves Itch with Minor Skin Irritations, **Sunburn** Relief, Bug Bites

❑ Shampoos:

- **Medicated shampoos**

o Shampoos are emulsions dosage forms used for hair and scalp cleansing which contain active ingredients designed to treat scalp conditions rather than just clean the hair.

o They are often recommended by dermatologists for issues such as dandruff, seborrheic dermatitis, and psoriasis.

o These shampoos can help relieve symptoms like itching, flaking, and inflammation E.g Ketoconazole shampoo.

Topical Emulsions

❑ Liniments

- Liniments are **alcoholic** or **oleaginous solutions** or **emulsions** of various medicinal substances intended to be **rubbed on the skin**

➢ The vehicle for a liniment should therefore be selected for **the type of action desired** (**rubefacient, counterirritant, or massage**) and also on the **solubility** of the desired components in the various solvents.

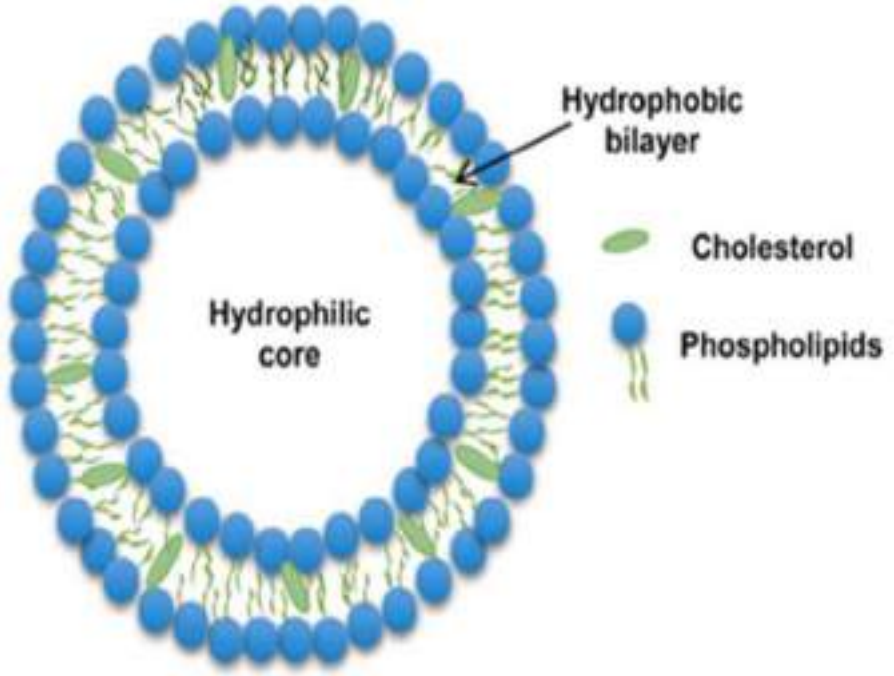
➢ All liniments should bear a label indicating that they are suitable only for external use and must never be taken internally.

➢ Liniments that are **emulsions** or that contain insoluble matter must be shaken thoroughly before use to ensure even distribution of the dispersed phase, and these preparations should be labeled shake well.

➢ liniments are prepared in the same manner as **solutions emulsions, or suspensions**

❑ Liposomes

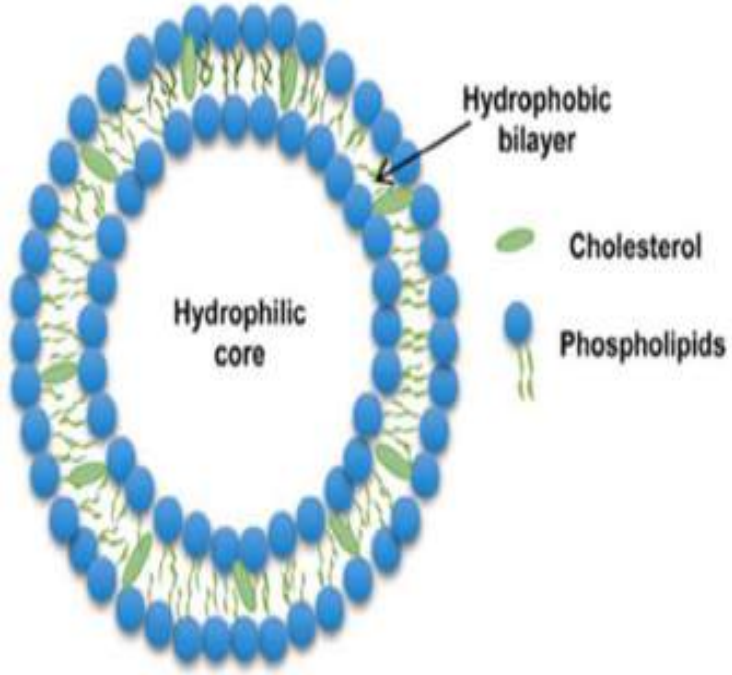
- **Liposomes** are the most explored nanocarriers used in **targeted** drug delivery systems.
- **Liposomes** are spherical lipid vesicles (usually 50–500 nm in diameter particle size) **composed** of one or more lipid bilayers, as a result of emulsifying natural or synthetic lipids in an aqueous medium
- Liposomes were first discovered in the 1960's by Bangham and later became among the most expansive drug delivery systems
- Liposomes nanoemulsions are widely used nanoparticles in nanomedicine mainly due to their biocompatibility, stability, ease of synthesis, high drug loading efficiency, high bioavailability, and the safe excipients used in these formulations.





❑ Liposomes

- Due to their size, hydrophobic and hydrophilic characteristics, and their ability to encapsulate drug molecules either in the aqueous interior of the vesicles or in the lipophilic membrane, liposomes are considered promising to be used effectively as drug delivery systems.
- Moreover, liposomes are suitable for diagnostic and therapeutic applications using several routes of administration, including ocular, oral, pulmonary, transdermal, and parenteral.
- Liposomes are primarily created from
  - **phospholipids** such as soybean phosphatidylcholine or egg yolk lecithin
  - **synthetic dialkyl or trialkyl lipids.**
- Although phospholipids can form bilayers on their own, these membranes are often **too fluid and unstable**, especially under physiological conditions.
- Therefore, the **incorporation of cholesterol is indispensable** for improving liposome stability.
  - Cholesterol is inserted **between the phospholipid molecules** in the bilayer. By doing so, it **regulates membrane fluidity**





اسئله وزاریه

Which of the following is true for the English or wet gum method of emulsion preparation?

a- The emulsifying agent is mixed with oil before adding water.
b- A mucilage of the emulsifying agent is prepared with water before adding oil.
c- It is used for volatile oils only.
d- The mixture is shaken in a capped bottle.
e- It is primarily for large-scale industrial production

In the context of emulsion stability, emulsion breaking is MOST likely to occur when:-

a- The emulsion is protected from light and microbial contamination.
b- The dispersed phase volume exceeds approximately 70% of the total volume.
c- An appropriate emulsifying agent is used at an optimal concentration.
d- Freezing and thawing
e- A and B

The preservatives commonly used in emulsions

a- Sodium lauryl sulfate
b- Alcohol in the amount up to 10%
c- Methylparaben and propylparaben
d- Magnesium stearate.
e- All.

Flocculation of emulsion means

a- Evaporation of internal phase
b- Evaporation of external phase
c- Phase separation
d- Aggregation or joining droplets together
e- Phase inversion

There are many terms for emulsion instability. Which term belongs to separation of dispersed droplets to form a layer of more concentrated emulsion?

a- Flocculation
b- Coalescence
c- Creaming
d- Cracking
e- Sedimentation

Emulsions may be prepared without inversion with about ____ of the volume of the product being internal phase.

a- 74%
b- 40%
c- 60%
d- 50%
e- 45%

Vegetable oil is used with limewater in nascent soap method for preparation of primary emulsion. Which vegetable oil is used with limewater?

a- Corn oil
b- Soybean oil
c- Coconut oil
d- Olive oil (Containing Oleic acid)
e- Peppermint oil

In continental method of preparation of emulsion, which statements below is INCORRECT?

a- The acacia or other o/w emulsifier is triturated with the oil.
b- Dry Wedgwood or porcelain mortar may be used.
c- After the oil and gum have been mixed, the two parts of water are added all at once.
d- The mixture is triturated immediately, rapidly, and continuously.
e- A mortar with a smooth rather than rough inner surface must be used.

Which of the following statements is True concerning forbes bottle method

a- It is suitable for volatile oils or oleaginous substances of low viscosities.
b- The dispersed phase is a mixture of fixed oil and volatile oil
c- Is suitable for fixed oils or oleaginous substances of low viscosities.
d- Is suitable for volatile oils or oleaginous substances of high viscosities.
e- None of them

In order to prepare a stable emulsion for small scale production: Which of the following is the right answer

A- rough surface mortar and pestle should be used
B- smooth surface mortar and pestle should be used
C- Colloid mill machine can be used
D- Passage of emulsion through a large homogenizer, in which the liquid is forced under great pressure through a small valve opening.
E- Large mixing tanks provided with jackets that permit heating and cooling of the ingredients and emulsion formed by the action of a high-speed impeller.

Unstable emulsion with cracking phenomena suffer from

a- the strong protective sheath about the globules of the internal phase.
b- It can be redispersed by shaking
c- It is separation of the internal phase from the emulsion.
d- It is irreversible
e- Both c and d.

Concerning dry gum method which of the following statements is True?

a- The acacia is triturated with the oil in a dry porcelain mortar.
b- Addition of internal phase to the external phase.
c- A mucilage of the gum is prepared by triturating in a mortar granular acacia with twice its weight of water.
d- The oil is added slowly to the mucilage of acacia in water.
e- Both a and b