

# EMULSIONS

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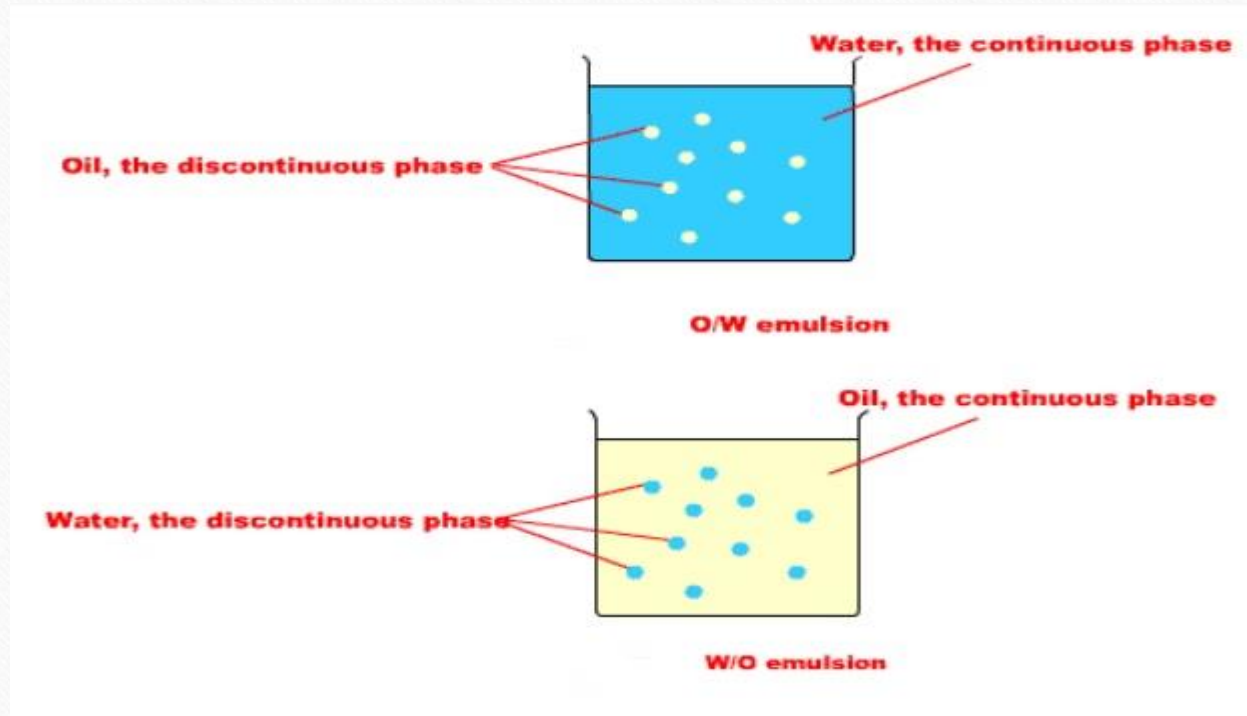
An emulsion is a thermodynamically unstable system consisting of at least two immiscible liquid phases one of which is dispersed as globules in the other liquid phase stabilized by a third substance called emulsifying agent.

OR

An emulsion is a dispersion in which the dispersed phase is composed of small globules of a liquid distributed throughout a vehicle in which it is immiscible.



The dispersed liquid is known as the Internal or Discontinuous phase. The droplet phase is called the dispersed phase whereas the dispersion medium is known as the External or Continuous phase. The liquid in which droplets are dispersed is called the external or continuous phase.



## **CLASSIFICATION**

- ❖ Based on type of emulsifying agent used
- ❖ Based on mode of administration
- ❖ Based on mode of dispersion
- ❖ Based on particle size
- ❖ Based on consistency
- ❖ Based on HLB

## **Based on type of emulsifying agent used**

- ✓ Emulsion containing natural gum – Acacia, Tragacanth
- ✓ Emulsion containing gum substitute – Cellulose & its derivatives
- ✓ Emulsion containing various soaps
- ✓ Emulsion containing saponins
- ✓ Emulsion containing starch
- ✓ Emulsion containing synthetic waxes – Emulsifying wax
- ✓ Emulsion containing natural waxes – Wool fat, bees wax
- ✓ Emulsion containing other emulsifying agent – Pectin, egg yolk.

## **Based on mode of administration**

- ✓ Oral use
- ✓ External use
- ✓ Parenteral use
- ✓ Rectal use

## **Based on mode of dispersion**

- ✓ O/W Emulsion
- ✓ W/O Emulsion
- ✓ W/O/W Emulsion
- ✓ O/W/O Emulsion

## **Based on particle size**

- ✓ Coarse emulsion ( < 5 micron)
- ✓ Fine emulsion ( > 5 micron)
- ✓ Micro emulsion ( nano )

## **Based on consistency**

- ✓ Liquid emulsion
- ✓ Semi solid emulsion

## **Based on HLB value**

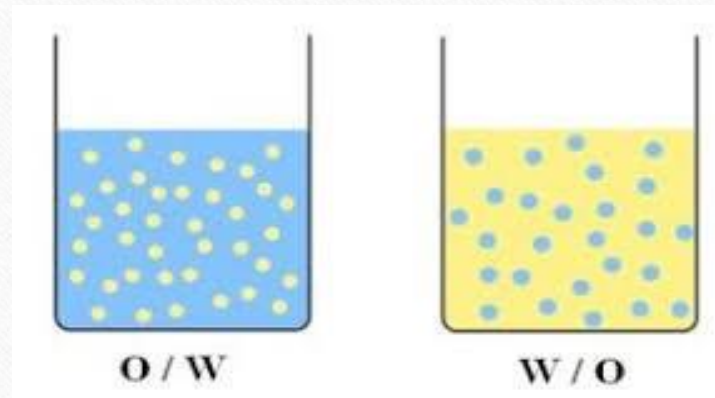
- ✓ Hydrophilic
- ✓ Lipophilic



## Types of emulsions

### O/W Emulsions

- Oil is dispersed phase
- Water is continuous phase
- Preferred for internal use
- Acacia, tragacanth, methyl cellulose – emulsifying agents



### W/O Emulsions

- Water is dispersed phase
- Oil is continuous phase
- Externally used emulsions
- Wool fat, resins, bees wax, soaps – emulsifying agents

## Identification tests

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- Dilution test
- Dye test
- Conductivity test
- Fluorescence test
- Cobalt chloride test

## Dilution test

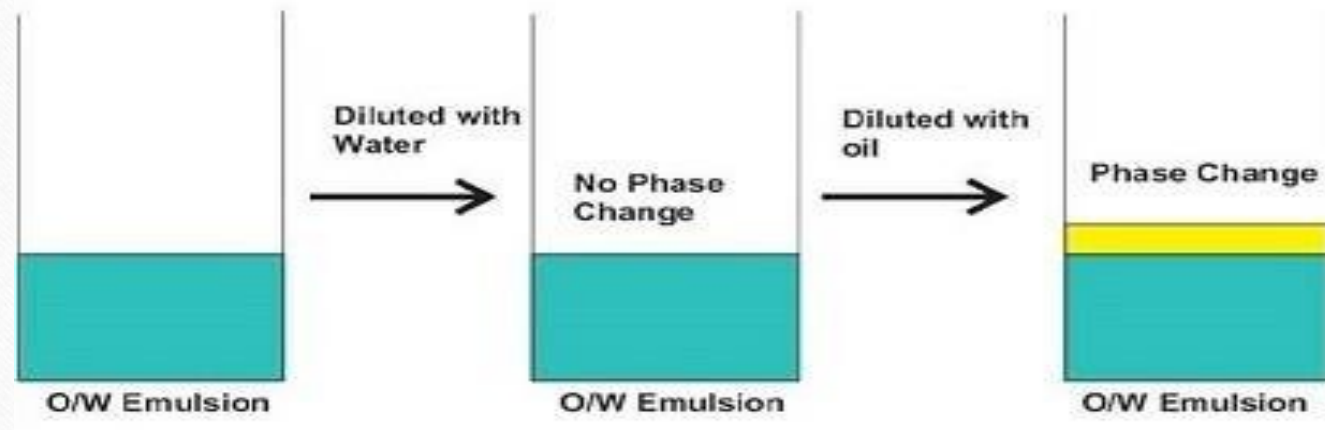
In this test the emulsion is diluted either with oil or water.

If the emulsion is o/w type and it is diluted with water, it will remain stable as water is the dispersion medium.

If it is diluted with oil, the emulsion will break as oil and water are not miscible with each other.

Oil in water emulsion can easily be diluted with an aqueous solvent

Water in oil emulsion can be diluted with a oily liquid.

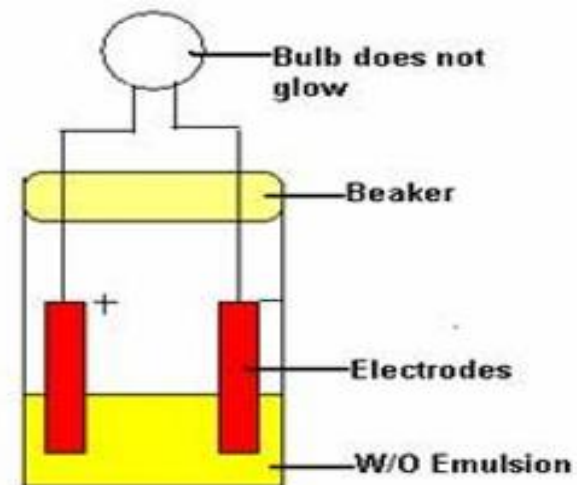
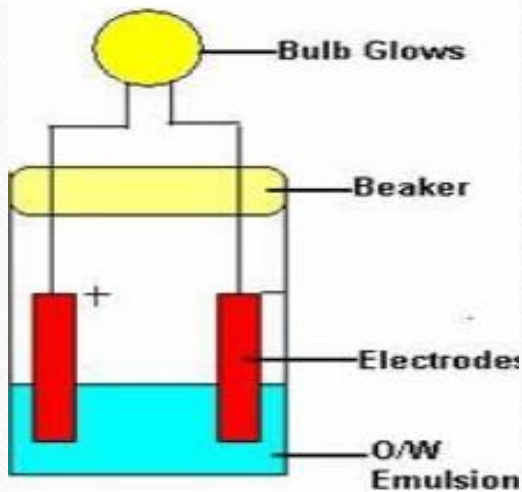


## Conductivity test

The basic principle of this test is that water is a good conductor of electricity.

Therefore in case of o/w emulsion, this test will be positive as water is the external phase.

‘In this test, an assembly is used in which a pair of electrodes connected to an electric bulb is dipped into an emulsion. If the emulsion is o/w type, the electric bulb glows.’



## Dye test

In this test an emulsion is mixed with a water soluble dye (amaranth) and observed under the microscope.

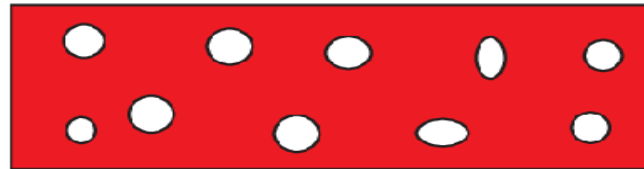
If the continuous phase appears red, it means that the emulsion is o/w type.

Water is in the external phase and the dye will dissolve in it to give color.

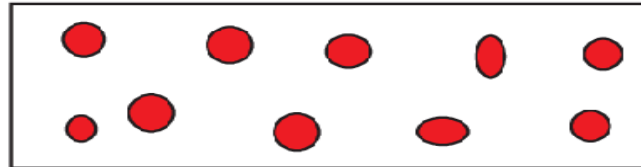
If the scattered globules appear red and continuous phase colorless, then it is w/o type.

Similarly if an oil soluble dye (Scarlet red C or Sudan III) is added to an emulsion and the continuous phase appears red, then it is w/o emulsion.

Water soluble dye



**O/W Emulsion**



**W/O Emulsion**

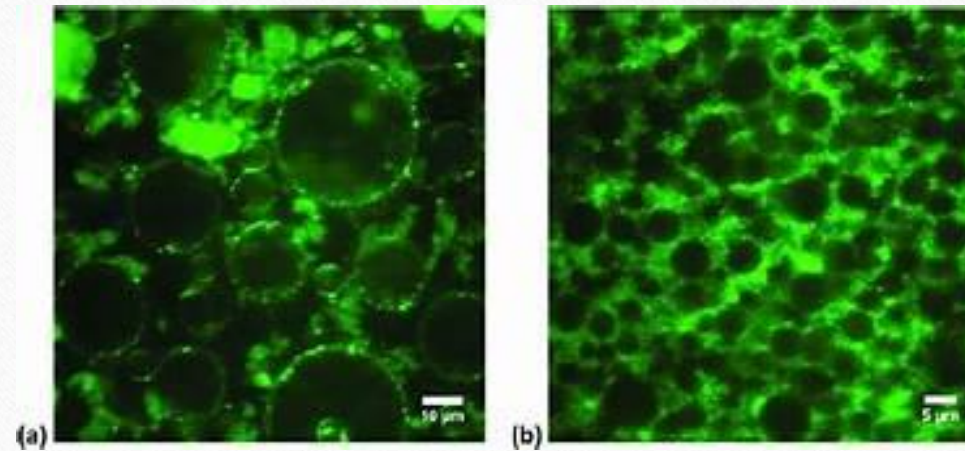
## Fluorescence test

Many oils show fluorescence when exposed to UV light.

If an emulsion on exposure to ultraviolet radiations

Whole field shows fluorescence under microscope, then it is w/o type

If it shows only droplets show fluorescence, then it is o/w type.



O/W Emulsion

W/O Emulsion

## Cobalt chloride test

Anhydrous cobalt is blue in color and hydrous cobalt is red or pink in color.

When a filter paper soaked in cobalt chloride solution

Dipped in to an emulsion and dried

if the color change occurs, from blue to red or pink

Preparation contains water as an external phase so, the emulsion is o/w type.



# FORMULATION OF EMULSION

- Emulsifying agents
- Preservatives
- Antioxidants
- Flavoring agents

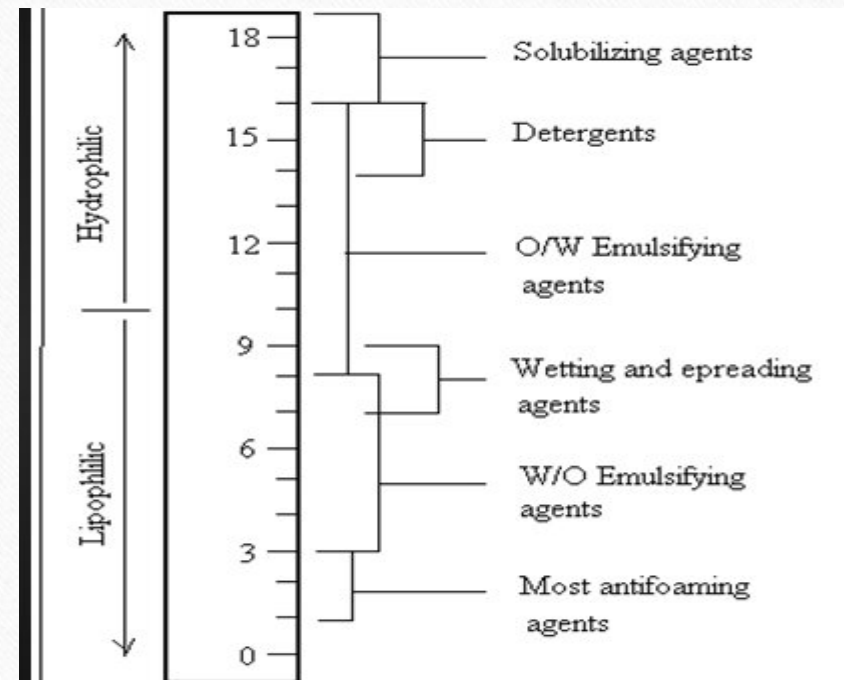


## Emulsifying agent

- ✓ Emulsifying agent reduce the interfacial tension between 2 phases that is oil phase & aqueous phase. Thus make them miscible with each other and form a stable emulsion.
- ✓ Emulsifying agents also known as emulgents or emulsifiers.
- ✓ Different emulsifying agents are available
- ✓ Difficult to select a proper emulsifying agent
- ✓ No single agent possess all properties
- ✓ Therefore sometimes two or more emulsifying agents are used to get a stable emulsion.

## HLB (hydrophilic lipophilic balance)

- ❑ It is derived by Graffin in 1949.
- ❑ Useful method for calculating balanced mixtures of emulsifying agents to provide particular type of emulsion
- ❑ HLB system characterize its relative polarity.
- ❑ Every emulsifying agent is given a number on H L B scale.
- ❑ More the hydrophilic interfacial barrier it favor o/w emulsion and while non polar barrier favor w/o emulsion.
- ❑ Oleic acid-1.8, Tween 80-15, SLS-40.



## Formulating with HLB value

- ❖ Mixing unlike oils together – H L B – 1-3
- ❖ Making W/O emulsion – H L B – 4-6
- ❖ Wetting powders into oils – H L B – 7-9
- ❖ Making self emulsifying oils – H L B – 7-10
- ❖ Making O/W emulsion – H L B – 8-16
- ❖ Making detergent solution – H L B – 13-15
- ❖ Solubilizing oil( micro emulsifying) – H L B – 13- 18

### **Qualities required for good Emulsifying agent:**

- It should reduce surface tension below 10 dynes/cm.
- It should be absorbed quickly on surface of dispersed medium.
- It should form coherent (non-adherent) film.
- It should be effective in low concentration.
- It should increase viscosity and maintain consistency.
- It should be compatible with other
- Non – toxic
- Chemically stable

## *Classification of emulsifying agents*

### Natural

Vegetable source : Acacia, tragacanth, starch, Agar, pectin, irish moss

Animal source : wool fat, egg yolk, gelatin

### Semisynthetic

Methyl cellulose, Na CMC

### Synthetic

Anionic: Sodium Lauryl Sulphate

Cationic: Cetrimide, benzalkonium chloride.

Non-ionic: Glyceryl ester- glyceryl monoesters

### Inorganic

Milk of magnesia, Mg oxide, Mg trioxide

### Alcohols

Carbowax, cholesterol and lecithin

## Natural emulsifying agents

### **Vegetable source:**

Carbohydrates

Gums & mucilaginous substances

Anionic in nature

Need preservatives for preparation of emulsion.

### **Acacia:**

Emulsion is attractive, palatable and stable in pH 2-5.

Preferred in o/w type of emulsion.

Low viscosity and form multimolecular film around globules

### **Tragacanth:**

Produce coarse, thick emulsion so rarely used.

Improve viscosity so used in combination with acacia

### **Pectin:**

1% mucilage in water used as EA.

It is incompatible with alkalis, strong alcohol, tannic acid and salicylic acid. iv. Starch:

It is rarely used as it forms coarse emulsion

**Agar :**

Not a good emulsifying agent

Produces very coarse & viscous emulsion.

Concentration of 2% is used.

**Irish moss (chondrus):**

Thickening agent.

Used in combination with acacia in cod-liver oil emulsion.

Concentration of 3% used as EA.

**Starch :**

Rarely used

Produce coarse emulsion

**Animal source:**

**Wool fat:**

Prepare external use emulsion, absorb 50% of water.

Produce o/w type of emulsion.

**Egg yolk:**

Prepare internal use emulsion.

Preservation in refrigerator.

It used concentration of 12 to 15%

**Gelatin:**

Used for emulsification of liquid paraffin emulsion.

It produce white and agreeable taste emulsion.

Concentration 1%

require preservation.



## Semisynthetic

### **Methyl cellulose:**

It is synthetic derivative of cellulose.

It is used as suspending/thickening agent.

Concentration of 2%.

It form precipitate with electrolytes.

### **Sodium Carboxy Methyl Cellulose:**

It is used as emulsion stabilizer.

It is used as suspending/thickening agent.

Concentration of 0.5 to 1.0 %.

## Synthetic

### **Anionic:**

These are used in the preparation of external use emulsion.

Ex. Alkali soap, metallic soap, sulphated alcohol, sulphonets.

Sodium lauryl sulphate is sulphonated alcohol produces o/w emulsion and form monomolecular film.

### **Cationic:**

Quaternary ammonium compound

e.g. benzalkonium chloride cetrimide.

Prepare external use preparation.

### **Non-ionic:**

Glyceryl mono stearate, sorbitan monopalmitate.

## Inorganic

### **Milk of magnesia:**

10 to 20%, prepare o/w coarse emulsion.

### **Magnesium oxide:**

5 to 10%, prepare o/w coarse emulsion.

### **Magnesium aluminium silicate:**

1% used to prepare o/w coarse emulsion.

### **Bentonite:**

5% is used to prepare o/w or w/o emulsion.

When used in o/w emulsion oil is added to the suspension of bentonite.

for w/o emulsion oil is placed in bottle and suspension of bentonite is added.

## Alcohols

### **Carbowaxes (polyethylene glycol):**

Used to prepare cream and ointment.

Molecular wt. 200-700 viscous, light colored, hygroscopic liquid.

Molecular wt. 1000 and above are wax solid.

### **Cholesterol:**

Ex. Cetyl alcohol, stearyl alcohol, cholesterol.

These are used to stabilize emulsion.

### **Lecithin:**

It form w/o emulsion.

They are rarely used as they darken the preparation.

## **Preservatives**

Number of ingredients in emulsion support the growth of microorganism which result into change in colour, odour and taste of emulsion.

Therefore there is need to include the preservative.

### **Sources of contamination:**

1. During the development or production.
2. Impure raw material.
3. Poor sanitation.
4. Improper ratio of o/w/gum.
5. pH change.

Examples :

Benzoic acid

Methyl paraben

Propyl paraben

Chloroform

Cetrimide.

## **Antioxidants**

Many organic compound subjected to autoxidation upon exposure to light and air and result on decomposition.

Unsaturated oils become rancid on autoxidation result in change in taste, odour and appearance.

Example:

Gallic acid, propyl gallate, ascorbic acid, butylated hydroxytoluene (BHT), butylated hydroxyanisol (BHA) in the range of 0.001 to 0.1%.

## **Flavoring agent**

Flavoring agent is incorporated in the formulation to impart the taste to it.

Example:

Vanillin – in liquid paraffin emulsion.

Benzaldehyde – in cod-liver oil emulsion.

## Methods of Preparation

- ❖ Dry gum method.
- ❖ Wet gum method.
- ❖ Bottle method.
- ❖ Other methods



### Preparation of primary emulsion:

Type of oils	Example	Ratio
Fixed oil	Castor oil	4:2:1
	Almond oil	
	Arachis oil	
	Cod-liver oil	
Volatile oil	Turpentine oil	2:2:1
	Peppermint oil	
	Cinnamon oil	
Mineral oil	Liquid paraffin	3:2:1



## Dry gum method

Calculate accordingly the ratio.

- ❖ Required quantity of oil in dry mortar
- ❖ Add required quantity of gum, triturate and mix uniformly
- ❖ Add required quantity of water and triturate vigorously
- ❖ Till primary emulsion is formed
- ❖ Primary emulsion – Clicking sound and white or nearly white color.

## Wet gum method

Calculate accordingly the ratio.

- ❖ Required quantity of gum in dry mortar
- ❖ Add required quantity of water, triturate and form a mucilage
- ❖ Add required quantity of oil in small portions and triturate vigorously
- ❖ Till primary emulsion is formed
- ❖ Primary emulsion – Clicking sound and white or nearly white color.



## Bottle method

- ❖ For volatile & non viscous oils
- ❖ Measure the required quantity of oil
- ❖ Add into a large bottle
- ❖ Add gum
- ❖ Mix thoroughly by shaking
- ❖ Add water
- ❖ Shake vigorously to form primary emulsion
- ❖ Add more water to produce the final volume

## Other methods

Blender & homogenizer

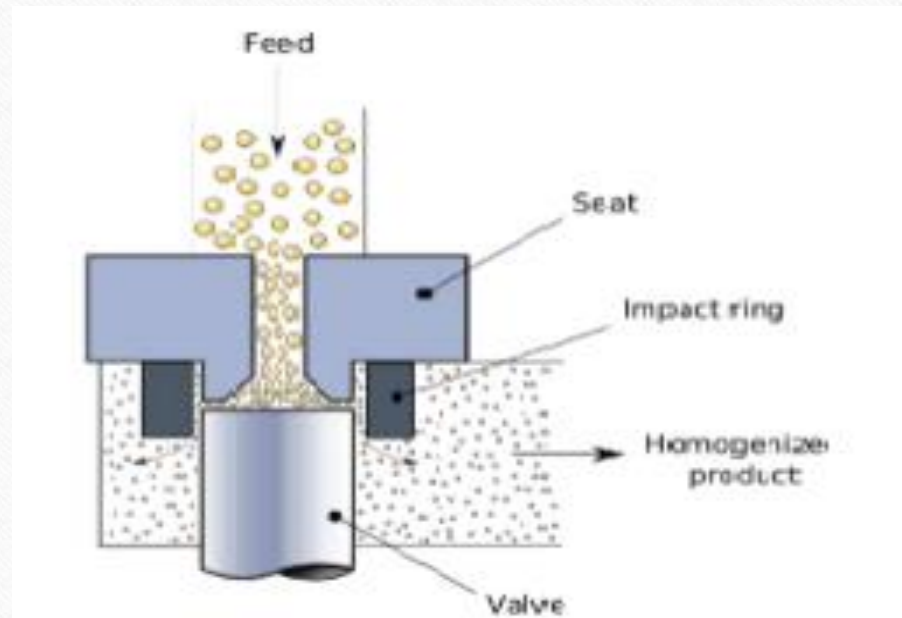
- Hand homogenizer
- Silverson mixer
- Colloidal mill

## Blenders & homogenizer principle

Large globules in coarse emulsion

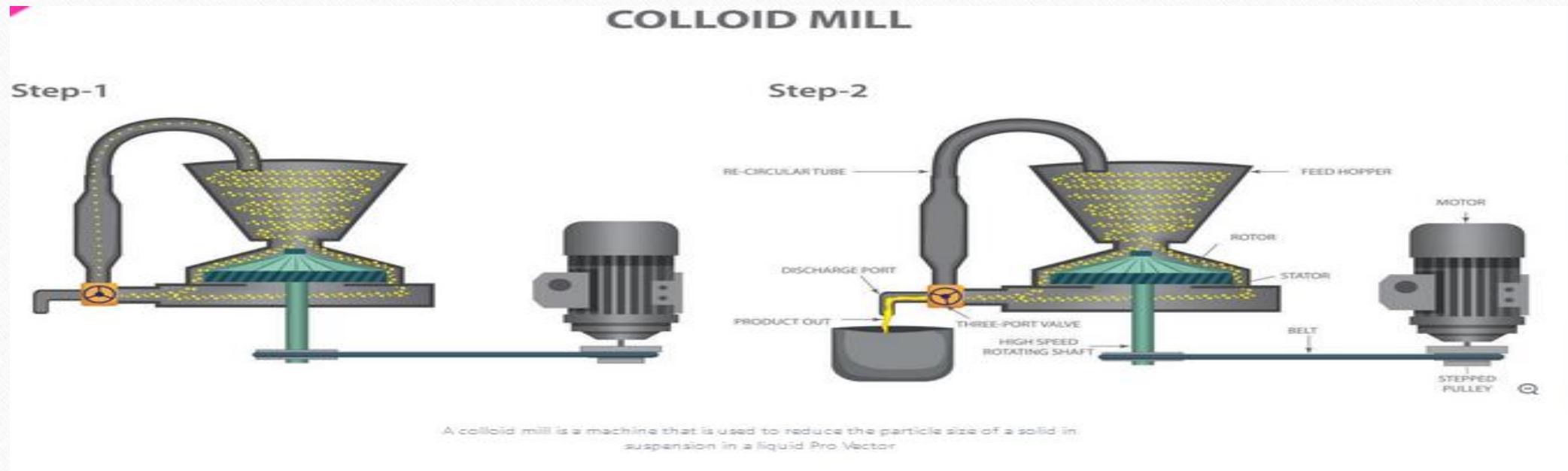
Under pressure through a  
narrow orifice

Smaller globules



## COLLOID MILL

- ❖ They operate on principle of high shear which is normally generated between rotor and stator of the mill.
- ❖ Colloid mill consists of a fixed stator plate and a high speed rotating rotator plate.
- ❖ Material drawn or pumped through an adjustable gap set between the rotor and stator, it is homogenized.
- ❖ The centrifugal force is created by high rotation of the rotor which operates within 0.005 to 0.010 inch of the stator.



## HAND HOMOGENISER

- ❖ In homogenizers the dispersion of two liquids is achieved by forcing their mixture through a small inlet orifice at big pressures.
- ❖ Homogenizers can be made with more than one emulsifying stage,
- ❖ it is possible to recycle the emulsion through the homogenizer more than one time.
- ❖ Homogenizers raise the temp. of the emulsion, hence cooling may be required.
- ❖ It can be used when a reasonably mono disperse emulsion of small droplet size ( 1 nm) is required.
- ❖ Hand operated



## SILVERSON MIXER

- ❖ Emulsified head covered with fine meshed stainless steel sieve
- ❖ Head consist of number of blades which rotate at very high speed
- ❖ Using electric motor at the top
- ❖ Head is placed in a vessel containing immiscible liquids
- ❖ Dipped into it
- ❖ Motor is started
- ❖ Liquids are sucked through the fine holes & oils is reduced into fine globules
- ❖ Fine emulsion is produced & then expelled out
- ❖ The intake & expulsion set up a pattern of circulation

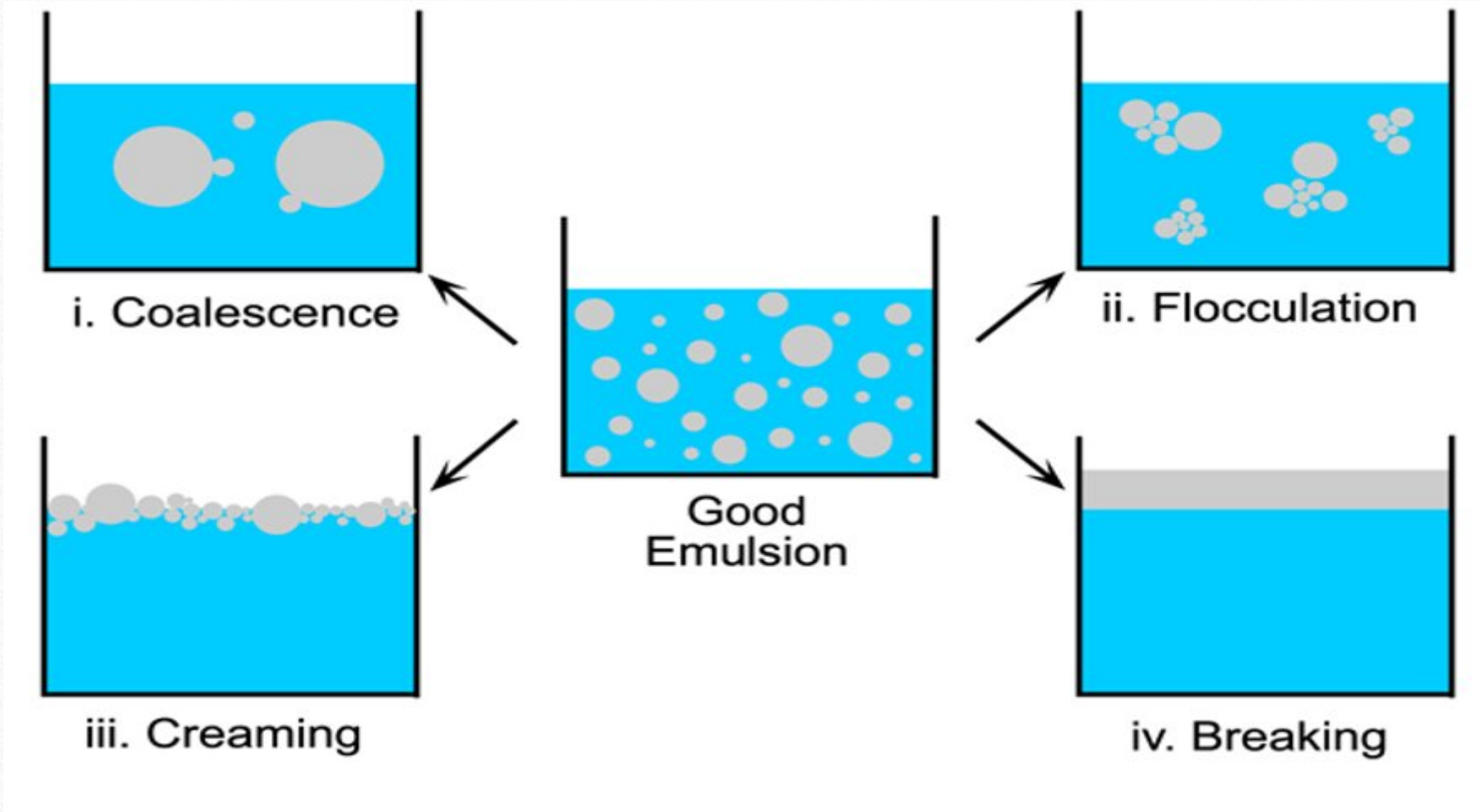
## Stability of emulsion

Garrett definition; A stable emulsion is one that would “maintain the same number of sizes of particle of the dispersion phase per unit volume of weight of the continuous phase”.

An emulsion is said to stable if it remains as such after preparation to its shelf life.

Following changes might be occurring:

- Creaming.
- Cracking.
- Phase inversion.





## Creaming

When large globules or aggregate of globules rises to the top of an emulsion or fall to the bottom and form concentrated thick layer.

Temporary phase.

Creaming should be avoided because it leads to cracking.

Stock law: 
$$V = \frac{2r^2 (d_1 - d_2) g}{9\mu}$$

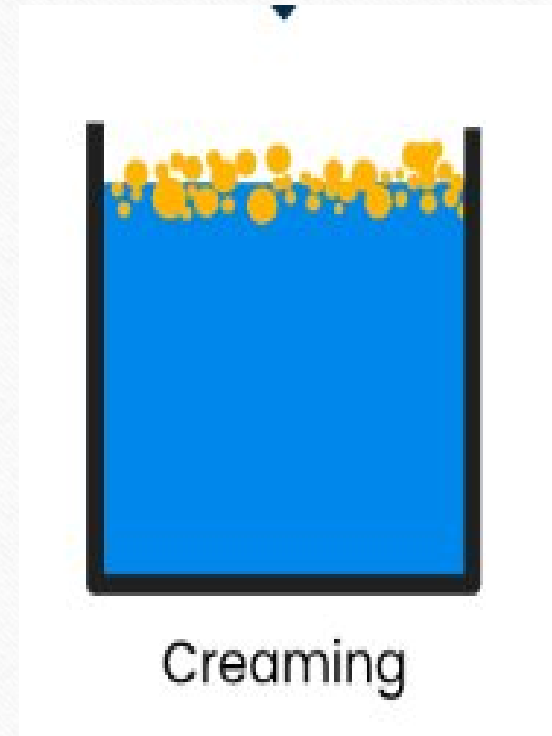
### Factor affecting creaming:

Radius of globules.

Density of dispersion medium/dispersing medium.

Viscosity.

Storage condition.



## Cracking

Greater destruction to an emulsion than creaming.

It is the coalescence of the globules of internal phase and separation of that phases in to a distinct layer.

This is irreversible, since the protective sheet above the globules of the internal phase no longer exist.

Re-stabilization by shaking is normally unsuccessful.

### **Reasons for cracking:**

Addition of opposite charged emulsifying agent.

Decomposition/precipitation of emulsifying agent.

Addition of common solvent.

Microorganism.

Change in temperature.

## Phase inversion

Phase inversion means change in the type of emulsion i.e. o/w to w/o or vice versa

### **Reasons for phase inversion.**

Addition of electrolyte.

Changing phase volume ratio.

Temperature change.

Changing the emulsifying agent.

## STORAGE & PACKAGING

- Depending on the use, emulsions should be packed in suitable containers.
- for oral use : usually packed in well filled bottles having an air tight closure.
- Light sensitive products : packed in amber colored bottles.
- For viscous emulsions : wide mouth bottles should be used.
- The label on the emulsion should mention that these products have to be shaken thoroughly before use.
- External use products should clearly mention on their label that they are meant for external use only.
- Emulsions should be stored in a cool place but refrigeration should be avoided as this low temperature can adversely effect the stability of preparation.



THANK YOU