

Formulation of Emulsion



Formulation of emulsions

Selection of Emulsifying agents (emulsifiers):

- An emulsifying agent is any material that enhances the stability of an emulsion (i.e. Prevention of coalescence and reducing creaming).
- The ideal emulsifying agent is colourless, odourless, tasteless, non-toxic, non-irritant and able to produce stable emulsions at low concentrations.

➤ Examples of Emulsifying agent

1. Carbohydrate Materials:

- Acacia, Tragacanth, Agar, Pectin. o/w emulsion.

2. Protein Substances:

- Gelatin, Egg yolk, Caesin o/w emulsion.

3. High Molecular Weight Alcohols:

- Stearyl Alcohol, Cetyl Alcohol, Glyceryl Mono stearate o/w emulsion, cholesterol w/o emulsion.


4. **Wetting Agents**

- Anionic, Cationic, Nonionic
- o/w emulsion
- w/o emulsion

5. **Finely divided solids**

- Bentonite, Magnesium Hydroxide, Aluminum Hydroxide o/w emulsion.

Test for identification of emulsion type

- Dilution test (miscibility test)
 - Staining test (dye solubility test)
 - Conductivity measurement
 - Fluorescence test
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THEORY OF EMULSIFICATION

- Film theory or adsorption theory
- Viscosity theory
- Wedge theory
- Interfacial tension theory

Film theory or adsorption theory

- As per this theory, the added emulsifying agent forms a mechanical film by getting adsorption at the interface of the liquid and offers stability to the emulsion. However, this theory could not explain the formation of type of emulsion.

Viscosity theory

- As per this theory, an increase in viscosity of an emulsion will lead to an increase in stability. This theory failed to explain about the milk which shows considerable stability even though its viscosity is less.

Wedge theory

- According to this theory, monovalent soap like sodium stearate give o/w type emulsion and divalent soap like calcium stearate give w/o type emulsion. This was explained by successful accommodation of the soap molecules to give the type of emulsion.


Interfacial tension theory

- In accordance with this theory the added emulsifying agent reduces the interfacial tension between the oil and water phases and thus a stable emulsion is formed. This theory could not explain the formation of type of emulsion.

The HLB (Hydrophilic lipophilic balance system) of emulsifying agents:

- An HLB number (1-20) represents the relative proportions of the lipophilic and hydrophilic parts of the molecule.
- High numbers (8-18) indicate a hydrophilic molecule, and produce an o/w emulsion.
- Low numbers (3-6) indicate a lipophilic molecule and produce a w/o emulsion.
- Oils and waxy materials have a 'required HLB number' which helps in the selection of appropriate emulsifying agents when formulating emulsions.
- Liquid paraffin, for example, has a required HLB value of 4 to obtain a w/o emulsion and 10.5 for an o/w emulsion.

HLB Scale

- HLB ca. 1 to 3.5: Antifoams
 - HLB ca. 3.5 to 8: Water-in-Oil Emulsifiers
 - HLB ca. 7 to 9: Wetting and spreading agents
 - HLB ca. 8 to 16: Oil-in-Water Emulsifiers
 - HLB ca. 13 to 16: Detergents
 - HLB ca. 15 to 40: Solubilizers
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HLB Values of Selected Emulsifiers

Chemical Designation	HLB Value
Ethylene glycol distearate	1.5
Sorbitan sesquioleate	3.7
Diethylene glycol monostearate	4.7
Sucrose diolate	7.1
Polyoxyethylene (4) lauryl ether	9.5
Polyoxyethylene (6) cetyl ether	10.3
Polyxyethylene (20) sorbitan tristearate	10.5
Polyxyethelene (9) nonyl phenol	13.0
Sodium Oleate	18.0
Polyxyethylene (100) Stearate	18.8
Potassium Oleate	20.0

Criteria For The Selection of Emulsifying Agents

- An ideal emulsifying agent should possess the following characteristics:
- It should be able to reduce the interfacial tension between the two immiscible liquids.
- It should be physically and chemically stable, inert and compatible with the other ingredients of the formulation.
- It should be non irritant and non toxic in the conc., used.
- It should be organoleptically inert i.e. should not impart any color, odour or taste to the preparation.
- It should be able to produce and maintain the required viscosity of the preparation.
- It should be able to form a coherent film around the globules of the dispersed phase and should prevent the coalescence of the droplet of the dispersed phase.

Preparation of Emulsion

The methods commonly used to prepare emulsions can be divided into two categories

A) Trituration Method

This method consists of dry gum method and wet gum method.

➤ Dry Gum Method

In this method the oil is first triturated with gum with a little amount of water to form the primary emulsion. The trituration is continued till a characteristic 'clicking' sound is heard and a thick white cream is formed. Once the primary emulsion is formed, the remaining quantity of water is slowly added to form the final emulsion.

This method consists of

"4:2:1" formula

4 parts (volumes) of oil

2 parts of water

1 part of gum

➤ **Wet Gum Method**

As the name implies, in this method first gum and water are triturated together to form a mucilage. The required quantity of oil is then added gradually in small proportions with thorough trituration to form the primary emulsion.

Once the primary emulsion has been formed remaining quantity of water is added to make the final emulsion.

This method consists of

"4:2:1" formula

4 parts (volumes) of oil

2 parts of water

1 part of gum



Mortar and Pestle



Homogeniser

B) Bottle Method

- This method is employed for preparing emulsions containing volatile and other non-viscous oils. Both dry gum and wet gum methods can be employed for the preparation.
- As volatile oils have a low viscosity as compared to fixed oils, they require comparatively large quantity of gum for emulsification.
- In this method, oil or water is first shaken thoroughly and vigorously with the calculated amount of gum. Once this has emulsified completely, the second liquid (either oil or water) is then added all at once and the bottle is again shaken vigorously to form the primary emulsion. More of water is added in small portions with constant agitation after each addition to produce the final volume.

Mechanical equipment for emulsification (Agitation)

- Mechanical stirrers
- Propeller type mixers
 - Turbine mixers
 - Homogenizers
- Colloid mills
- Ultrasonifiers




Mechanical stirrers




Colloidal mill

Stability of Emulsion

- An emulsion is said to be stable if it remains as such after its preparation , that is the dispersed globules are uniformly distributed through out the dispersion medium during its storage. The emulsion should be chemically stable and there should not be any bacterial growth during it shelf life.
 - Emulsion instability may either reversible or irreversible and manifest in the following ways:-
 - 1) Cracking (irreversible instability)
 - 2) Flocculation
 - 3) Creaming
 - 4) Phase inversion
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1) Cracking:-

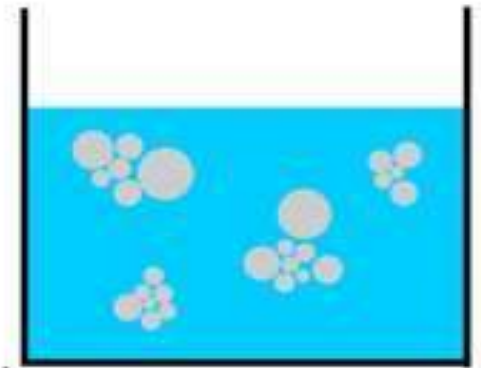
- ▶ Cracking means the separation of two layers of disperse and continuous phase , due to the coalescence of disperse phase globules which are difficult to redisperse by shaking.
 - ▶ Cracking may occurs due to following reasons:-
 - i. By addition of emulsifying agent of opposite type
 - ii. By decomposition or precipitation of emulsifying agent
 - iii. By addition of common solvent
 - iv. By microorganisms
 - v. Change in temperature
 - vi. By creaming
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i. Coalescence



Good Emulsion



ii. Flocculation




iii. Creaming



iv. Breaking

2) Flocculation :-

- ▶ In flocculated state the secondary interaction (van der waals forces) maintain the droplets at a defined distance of separation.
 - ▶ Application of shearing stress to the formulation (shaking) will redisperse these droplets to form a homogeneous formulation.
 - ▶ Although flocculation may stabilise the formulation, there is also possibility that the close location of droplets would enable droplet coalescence to occur if the mechanical properties of the interfacial film are compromised.
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3) Creaming:-

- ▶ Creaming may be defined as the upward movement of dispersed globules to form a thick layer at the surface of emulsion.
- ▶ Creaming is temporary phase because it can be re-distributed by mild shaking or stirring to get again a homogenous emulsion.
- ▶ The factors affecting creaming are described by stoke'TMs law:

$$V = \frac{2r^2 (d_1 - d_2) g}{9\eta}$$

- ▶ Where V= rate of creaming
- ▶ r=radius of globules
- ▶ d_1 = density of dispersed phase
- ▶ d_2 = density of dispersion medium
- ▶ g= gravitational constant
- ▶ η = viscosity of the dispersion medium

The following approaches can be used for decreasing Creaming

- a. Radius of globules
- b. Difference in density of disperse phase and continuous phase
- c. Viscosity of dispersion medium
- d. Storage condition

4) Phase inversion:-

- Phase inversion means the change of one type of emulsion into other type, that is oil in water emulsion changes into water in oil type and vice-versa.

Due to following reasons the phase inversion takes place:-

- i. By the addition of an electrolyte
- ii. By changing the phase-volume ratio
- iii. By temperature change
- iv. By changing the emulsifying agent

▶The phase inversion can be minimised by keeping concentration of disperse phase between 30 to 60 % , storing the emulsion in cool place and using a proper emulsifying agent in adequate concentration.