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Subject: Food Technology

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Paper No. : 04 Unit Operations in Food Processing

Module : 31 Size Reduction-2: Emulsification and Homogenization

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Description of Module	
Subject Name	Food Technology
Paper Name	04 Unit Operations in Food Processing
Module Name/Title	Size Reduction-2: Emulsification and Homogenization
Module Id	FT/UOFP/31
Pre-requisites	-
Objectives	To enable students to select a proper homogenization device for different food use by giving them sufficient indepth knowledge on the principle and operation of homogenizer.
Keywords	Homogenization, emulsion, power density, homogenization efficiency

31.1 Introduction

So far we have discussed about the size reduction of solid food materials and the machinery associated with the size reduction of solid food materials. In this module we shall discuss about the size reduction of food components in liquid form. There are various techniques by which the immiscible liquids or solid-liquid are been able to kept in dispersed form. Emulsion is the mixture of two liquid or solid-liquid components which are in dispersed phase i.e. they are thoroughly mixed to each other; however, they are not chemically bonded. The process by which emulsion is formed is called emulsifying or emulsification. We shall discuss the emulsification procedures in subsequent sections. Homogenization is the process of breaking of food components such that they form a stable emulsion. We shall discuss the mechanism of homogenization, types of homogenizers used in food industries, application of homogenization and the effect of homogenization in final product in this module.

31.2 Emulsification

Emulsification is the process by which two immiscible liquids or solid-liquid can be dispersed one into the other by severe mixing, stirring and beating. Most often, in food system we see immiscible liquids like water and oil. Milk is a very common example of emulsion of water and oil. In emulsion we see that one liquid is in dispersed phase and the other one in continuous phase. The character of emulsion is mainly determined by its continuous phase. Based on the phases, emulsions are categorised into two types. One is oil in water (O-W) emulsion and the other one is water in oil (W-O) emulsion.
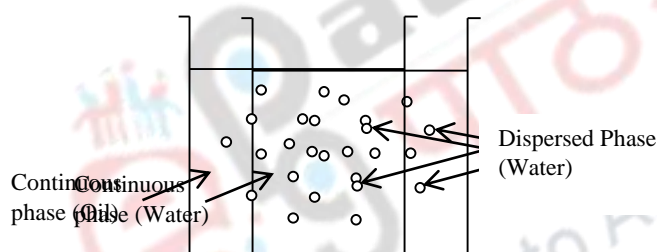

Fig.31.1 (b) W-O Emulsion   ion

Examples: O-W emulsion: milk, cream, mayonnaise, ice cream

W-O emulsion: butter

31.2.1 Mechanism of emulsification

In food product development, many food emulsions are developed combining two or more liquids which are not miscible to each other. To characterize the food emulsion in terms of physicochemical and sensory properties one need to know the concentration of ingredients the raw material contains, the droplet size of the immiscible components so that a proper method of emulsifying can be chosen. There are many factors which are responsible for the stability of emulsion. They are;

  i.   interfacial tension between the two phases,
  ii.   size of the dispersed phase droplets,

iii. viscous properties of continuous phase, and
iv. density difference between two phases
v. type and quantity of emulsifying agents

The stability of an emulsion depends on the shape of the droplets; an irregular shaped droplet has more surface area to interact with the other droplet and higher affinity to coalesce. In contrary, a spherical droplet minimizes the surface area. The interfacial forces responsible for making the droplet in to spherical form is characterized by Laplace pressure (ΔP_L) which acts across the oil-water interface toward the centre of droplet so that the inside droplet pressure is higher than that of outside pressure.

$$\Delta P_L = \frac{4\gamma}{D} \qquad \dots (31.1)$$

Where, γ is the interfacial tension between oil and water, D is the droplet diameter. The equation indicates that, the pressure required to disrupt a particle increases with increase of Laplace pressure or interfacial tension between the two phases and decrease of droplet diameter. So, an emulsion is able to get stabilised when the interfacial tension is less. The continuous phase's viscosity plays a major role in the way the dispersed particles deform. Higher the viscosity of continuous phase, higher is the shearing action imparted on the droplet. The relationship between the shear stress and interfacial tension is characterised by a number called Weber number (W_e).

$$W_e = \frac{Shear\ force}{Interfacial\ tension} = \frac{s^{-1}\mu D}{2\gamma} \qquad \dots (31.2)$$

Where, s^{-1} is the shear rate, μ the viscosity, D the diameter of droplet and γ the interfacial tension. A critical Weber number be defined for a particular system where the values are just to disrupt. A Weber number above the critical value means droplet disrupts. In other words, if the shear rate is more or diameter of particle is more, there is acute chance of disruption. Coalescence of particles is hindered by increasing viscosity of the continuous phase. The lesser the density difference between the two phases $(\rho_w - \rho_o)$, lesser is the chance of separation. The physical stability of an emulsion is governed by Stoke's law.

Fig.31.2 Profile of droplet disruption due to shear force

$$v = \frac{gD^2(\rho_w - \rho_o)}{18\mu} \qquad \dots (31.3)$$

As a whole, the stability of emulsion is a described by the lower density differences, lower interfacial tension and higher viscosity of continuous phase. The disruption force required can be found out by Weber number.

31.2.2 Emulsifiers

Since, the dispersed droplet tend to coalesce, most of the emulsions cannot remain in stable form and eventually separates from each other. Addition of a third substance which makes the emulsion stable is called emulsifier. Emulsifier reduces the interfacial tension between the droplets and help to create large surface area of the dispersed phase by forming a protective coating around the droplet. The characteristics of emulsifier can be explained by its
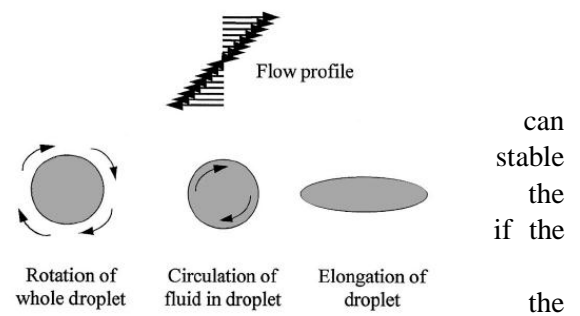
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bi-polar nature viz. polar and non-polar groups. The polar group (hydrophilic) orients towards the water phase and the non-polar groups (lypophilic) orients towards the oil phase, thus makes an inter-link between the two phases by not allowing them to coalesce.

The most common examples of emulsifiers are lecithin, phosphate, glycerol monostearate, etc.

31.3 Homogenization

Many food systems are prepared from the two phases viz. oil and water. Vitamins, colorants, surfactants etc. are mixed in water phase of food to form a food system. Likewise protein, polysaccharides, sugars, salts etc. are mixed with oil phase. The process of converting separate oil and water phases to a stable emulsion is called *homogenization*. The mechanical device used to prepare the emulsion is called *homogenizer*. Basically, homogenization is categorized into primary homogenization and secondary homogenization based on the nature of the starting material. If the emulsion is formed directly from the two separate liquids, the process is called *primary homogenization*. Preparation of salad dressing by mixing aqueous phase and oil is an example of primary homogenization. In an existing emulsion, if the droplet size of the dispersed phase is further reduces, the process is called secondary homogenization. In raw milk, fat globule size is reduced to less than 1 μm to form a stable emulsion. This is an example of secondary homogenization. In most food processing operations, the secondary homogenization is mostly done to form a stable emulsion where, the separate oil and water phases are made in to a course emulsion followed by further reduction of the droplet size.

The two separate phases oil and water tend to be in a thermodynamically stable state where the contact area between the two phases is minimum. Since, the density of oil is less than that of aqueous phase, oil form the top layer. To make an emulsion, the liquids are shaken vigorously so that the oil phase disrupts and intermingled with water phase. The oil droplets formed continuously move around and coalesce with other droplets, thus forming larger droplets. As this process continues, the larger droplets move upward and form a separate layer. As a consequence, the emulsion reverts back to the   original form. The hydrophobicity of oil is making the system stable.

31.3.1 Mechanism of homogenization

The fat globule passes through a narrow slit, whose diameter is just larger than that of the fat globule. Due to high pressure and narrow slit a shear force is developed which acts in opposite direction of the flow. The surface of the globule becomes wavy and eventually breaks into droplets. Intense research has been done on the mechanism of homogenization, however, exact mechanism is not known. Micro turbulence and cavitation are the two most accepted mechanism of homogenization.

Turbulence

Turbulence occurs when flow rate of fluid exceeds a critical limit. Small eddies are generated due to turbulence which is characterised by rapid and chaotic fluctuations of velocity of fluid. The eddies thus formed disrupt the oil droplets due to its large shear and pressure gradient. The size of the eddy depends on the velocity of flow and the viscosity of the liquid. The smaller the size of the eddy, higher is the shear and pressure gradient. So, it is interpreted that larger eddies are less effective than the smaller eddies. However, very small eddies are also ineffective because maximum energy is dissipated through viscous losses rather than through droplet disruption. For isotropic turbulent condition, the Weber number is given by;

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$$W_e = \frac{Turbulent\ force}{Interfacial\ force} = \frac{C\ \rho_c^{1/3} \varepsilon^{2/3} d^{5/3}}{4\gamma} \qquad (31.4)$$

Where, C is a constant related to critical Weber number, ρ_c is the density of continuous phase and ε is the power density. Under isotropic turbulent condition, the maximum size of droplet that can persist during homogenization can be from equation (31.4) and given as follows;

$$d_{max} = \frac{C'\gamma^{3/5}}{\rho_c^{1/5}\varepsilon^{2/5}} \qquad (31.5)$$

Where, C' is a constant which depends on the characteristic dimensions of the system. This equation indicates that the size of the droplets produced under turbulent conditions decreases as the power density increases, the interfacial tension decreases or the density of continuous phase increases. Viscosity has no direct relation with the droplet size; however, if the density ratio $\frac{\mu_d}{\mu_c}$ falls within 0.1 and 5, the maximum possible droplet size which can persist homogenization pressure can be obtained.

Cavitation

Cavitation is a phenomenon occurs when there is a drastic pressure changes. These kinds of pressure changes are often seen in case of ultrasonic and high pressure homogenizers. The fluid contracts when the pressure acting on it increases and it expands when the pressure decreases. When the instantaneous pressure falls below a critical value, cavity is formed. As the fluid expands due to low pressure, the cavity grows. The surrounding liquid becomes thermodynamically unstable and evaporates and moves into it. During a subsequent compression, the cavity suddenly collapse which generates a shock waves. The shock waves propagate to the surrounding liquid and droplets of the near vicinity disrupt and disintegrate. Though high pressure and temperature prevail near the cavity, the phenomenon is localized and occurs instantaneously, any physical damage to the homogenizer is avoided. The cavitation pressure of fluid depends on the type of fluid and the air content of the fluid. This pressure is known as *cavitational threshold*. The cavitational threshold depends on the frequency of pressure fluctuation occurring during homogenization.

31.3.2 Homogenizers

High speed blender

The most common type of homogenizing device used in food industries are high speed blenders. The blender consists of a high speed motor which rotates a shaft inside a vessel. The rotation of the shaft generally varies from 20 to 2000 rpm. The blades attached to the shaft generate longitudinal, rotational and radial motion of the fluid causing disintegration of the droplets of the two phases. Longitudinal velocity profile increases the efficiency of homogenization. This can be achieved by placing baffles on the wall of the vessels. There are various designs of the blades available to suit the varied product profile. High speed blenders are suitable to homogenize intermediate or low viscosity products. The droplet size decrease as the homogenization time increases and reaches a critical minimum diameter beyond which no further reduction takes place. The critical droplet size achieved in high speed blender depends on the nature and concentration of the ingredients used. Typically, the droplets produced by high speed blenders vary between the diameter 2 to 10 µm. The energy density is one among the lowest of all the homogenization devices available.

Colloid mill

Colloid mill is suitable for homogenization of medium and high viscosity food products. Basically, the colloid mill gives more efficient results when the premixed emulsion is fed into it. In other word, we can say, secondary homogenization is done in colloid mill. The coarse emulsion formed by high speed blender is fed to the mill which passes through the narrow gap between two discs viz. *rotor* (rotating disc) and *stator* (static disc). The rotation of the rotor generates shear stress in the gap between the discs and disintegrates the larger droplets to fine droplets. The intensity of stress produced can be varied by adjusting the gap between the discs (50 to 1000 μm). Typically, the rotational speed of the rotor varies between 1000 and 20000 rpm and the droplet size achieved varies from 1 to 5 μm. The finer droplets can be obtained by allowing the emulsion in the gap for a longer period of time. This can be done by reducing the flow rate of the emulsion or by passing the emulsion through the mill number of times. However, by doing so, the manufacturing costs increases. Cooling arrangement should be provided to reduce the temperature of the device caused due to the viscous dissipation energy losses.
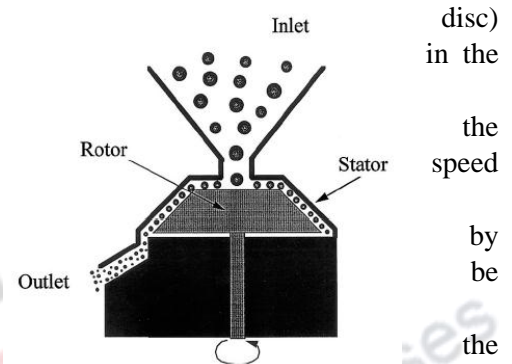

Fig.31.3 Schematic of a Colloid mill

High pressure valve homogenizer

High pressure valve homogenizers are suitable for homogenizing premixed emulsion of small droplets. The most common food that is homogenized in this homogenizer is milk. As the name suggests, it has a valve and seat with a narrow gap between them. A piston pump sucks the liquid in its back stroke and pushes to the narrow gap between the valve and seat in the forward stroke. During the passage through the gap, the droplets encounter high shear, cavitational and turbulent condition as a result of which the coarse droplets break into number of fine droplets. For different products, the valve design may vary. The valves are spring loaded so that the gap between the valve and seat can be varied (typically between 15 and 300 μm). Decreasing the gap, the pressure drop across the valve increases, as a result fine droplets are produced. But, at the same time the input energy requirement increases. If we draw a graph between the pressure and droplet size, we obtain a linear curve. The typical throughput of industrial homogenizers varies between 100 and 20000 l/h and the pressure variation between 3 and 20 MPa. As per the applicability of final product, some commercial homogenizers use "two-stage" process where the product passes through two valves arranged in series. The pressure applied to the first valve is more than the pressure of the second valve, because least energy is required to break the clumps formed after first homogenization. Using this method, very small size droplets usually less than 1 μm can be achieved. High pressure homogenizers are able to produce a variety of different food products, although they are more suitable for low and intermediate viscosity products.
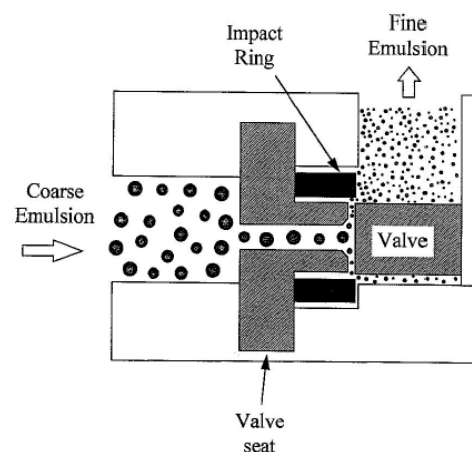

Fig.31.4 High Pressure valve homogenizer

Ultrasonic homogenizer

Ultrasonic homogenizers are relatively latest invention which uses ultrasound to product emulsion. High intensity ultrasonic waves are produced which generate intense shear and pressure gradient within the product that disrupt the droplets mainly due to cavitational effect. The two common methods used to produce ultrasonic waves are piezoelectric transducers and liquid jet generators. Piezoelectric transducers are commonly used with laboratory homogenizers that are ideal for preparing

Fig.31.5 Ultrasonic homogenizer with liquid jet generator

small volume of emulsion. The transducer consists of a piezoelectric crystal contained within a protective metal casing, which is tapered at the end. A high intensity piezoelectric wave is applied to the transducer which oscillates at high speed and generates an ultrasonic wave. The ultrasonic wave is directed towards the tip of the transducer which then radiates into the surrounding liquid. It generates intense pressure and shear gradients (mainly due to cavitational effect) which causes the liquid to be broken into small droplets and intermingled with each other.
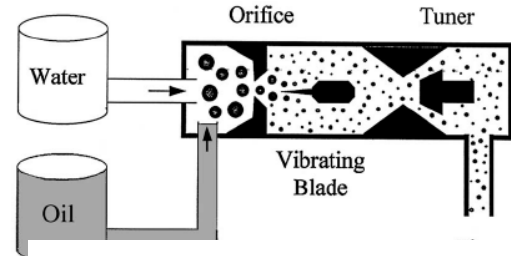
A stream of fluid is forced to the sharp edged blade, which causes the blade to vibrate rapidly and generates ultrasonic waves. The droplets in the near vicinity are broken up due to high shear, cavitation and turbulence. The advantage of ultrasonic homogenizer is that it can produce continuous emulsion and more energy efficient that high pressure valve homogenizer. The efficiency of this homogenizer depends on the intensity, duration and frequency of the ultrasonic waves. The homogenization frequency decreases with increased frequency. Most commercial devices use a frequency range from 20 to 50 kHz. The droplet size can be reduced by increasing the intensity or duration of the ultrasonic radiation.

Microfluidizer

Emulsions from individual phases with very small droplet size are formed in a microfluidizer by allowing the phases to accelerate at high velocity and then impinging them on a surface. The impinged droplets intermingled with each other and form stable emulsion. Very fine droplets can be formed by allowing the emulsion through the microfluidizer number of times.
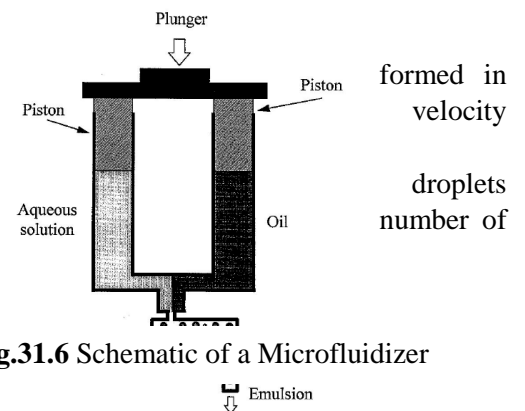

Fig.31.6 Schematic of a Microfluidizer

Membrane homogenizer

The dispersed phase is forced to the continuous phase through a membrane with definite pore size. The size of the droplet depends on the pore size of the membrane and the interfacial tension between the two phases. The membrane can be manufactured with varying pore size and it has to be strong to withstand high pressure. The membrane technique can be applied either in batch process or continuous process. In the batch process, the dispersed liquid is forced through a cylindrical membrane dipped in a continuous phase which is kept in a vessel. In the continuous phase process, the continuous phase passes through a cylindrical membrane and the dispersed phase is forced to the continuous phase through small tubes. The major advantage of membrane homogenizer is that it can produce a narrow range of droplet size distribution. The energy efficiency is excellent as no energy is dissipated through the viscous losses.
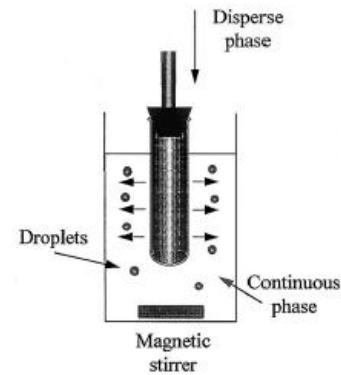

Fig.31.7 Batch type membrane homogenizer

31.3.3 Homogenization Efficiency

The efficiency of homogenization is calculated by comparing the actual energy required to form an emulsion and the minimum theoretical energy required.

$$\eta_H(\%) = \frac{\Delta E_{min}}{\Delta E_{total}} \times 100 \qquad (31.6)$$

$$\Delta E_{min} = \Delta A \gamma \qquad (31.7)$$

Where, ΔE_{min} is the minimum energy required to form an emulsion, γ is the interfacial tension between oil and water phase, ΔA is the total increased area during homogenization and ΔE_{total} is the actual amount of energy expended during homogenization. The efficiency of homogenization is less than 1% because to disintegrate small droplets huge pressure gradient is required, that should be equal to or higher than Laplace pressure gradient ($\approx 2\gamma/r^2$). So, homogenizers are the most energy inefficient devices. However, the efficiency can be improved by reducing the pressure gradient which can be accomplished by reducing the interfacial tension) or by minimizing the viscous dissipation.

31.3.4 Factors affecting droplet size

The main factors affecting droplet size in homogenization are:

1. *Type of homogenizer*: Variation in the design of homogenizer valve changes some parameters like power density $\varepsilon = \Delta P/t_p$ which has a direct relation with induced stress for droplet disruption.

2. *Homogenizing pressure*: From the relation, $\propto P^{1.5}$, we interpret that the power density has a positive relationship with the homogenization pressure. So, increasing the homogenization pressure, the droplet size formed reduces. But after certain size, no further decrease in size of droplet happens with further increase in homogenization pressure.

3. *Stages of homogenization*: Size reduction of droplets mainly a function of first stage homogenization. The second stage homogenization has least effect on the size reduction. It only breaks the fat clumping which is a common tendency of fat seen after first stage homogenization.

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4. *Dispersed phase and the surfactants*: The proportion of dispersed phase has a significant effect on the size of droplet formation. More the dispersed phase present more surface area is created. So, sufficient surfactant must be present to form new droplet membrane.

5. *Type of surfactants*: Surfactants of different nature yield different interfacial tension. As a result the droplet size varies. As for example low molecular weight surfactants like Tween-20 or sodium dodecyle sulphate create low effective interfacial tension and so smaller globule results.

6. *Temperature*: Before homogenization the fat should be in liquid state. The homogenization efficiency reduces if the fat is in crystalline state. So, homogenization is done at about 50 ℃.

7. *Proper operation of homogenizer*: Pressure fluctuation, worn homogenizing valve, and air inclusion adversely affect the droplet size formation.

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