Consortium for Educational Communication

Module on

Centrifugation

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Text

Introduction

Foods are complex mixtures of compounds and the extraction or separation of food components is fundamental for the preparation of ingredients to be used in other processes (for example cooking oils from oilseeds or gelatin from connective tissue); or for retrieval of high value compounds, such as enzymes (e.g, papain from papaya for meat tenderization or rennet from calf stomachs for cheese making) etc.

There are three main categories of separation:

1. Separation of liquids and solids from slurries, pastes, particulates or flours, where either one or both components may be valuable (for example juices, pectin, enzymes, cooking oil, cream and coffee solubles).

2. Separation of small amounts (less than 2%) of solids from liquids. Here the main purpose is purification of water or clarification of liquids such as wine, beer, juices, etc. and the solids are not valuable.

3. Extraction of small amounts of valuable materials using a solvent. Different methods are used to achieve any one of the objective mentioned above and centrifugation is one of them. Generally, centrifuges are used throughout many manufacturing industries (Table 1), to separate suspended solids from liquid utilizing the centrifugal acceleration of the suspended particles directed outward from the axis of rotation. This force initiates the particle movement to the centrifuge periphery where

it is trapped or contained by the wall of the rotating body. Alternatively, a density difference between two immiscible liquids is exploited to accelerate separation of the liquids (i.e. fat separation in dairies for cream or butter manufacture). The most common application is the separation of solid from highly concentrated suspensions, which is used in the treatment of sewage sludges for dewatering where less consistent sediment is produced. In the chemical and food industries, special centrifuges can process a continuous stream of particle-laden liquid. There are two main applications of centrifugation:

1. Separation of immiscible liquids and separation of solids from liquids.

2. Separation of solid particles from air by centrifugal action is done in the 'cyclone' separator.

A specialized use involves separation of water from fresh-cut vegetables before modified atmosphere packaging.

Table. 1. Industrial use of centrifugal technologies

Food and agri-business

Sugar crystal recovery

Dewatering of fresh-cut salad and vegetables

Milk processing, bacterial removal, cream separation

Pulp-free orange juice

Formation of fruit and vegetable juices

Frying oil clean-up

Pharmaceutical/biotechnology

Recovery of valuable isolates

Recovery of cells (yeast and bacteria, plant and animal cells)

Clarification of fermentation broths

Environmental industries

Sewage solids recovery

Wastewater treatment

Removal of metal cuttings from industrial cutting lubricants

Chemical industries

Black coal separation from slurries

Isolation of synthetic products

Gas-phase

Centrifugation

Centrifugation is a process which involves the application of the centripetal force for the sedimentation of heterogeneous mixtures with a centrifuge, and is used in food industry and laboratory settings. This process is used to separate two miscible substances, but also to analyze the hydrodynamic properties of macromolecules. More-dense components of the mixture migrate away from the axis of the centrifuge, while less-dense components of the mixture migrate towards the axis. Food Chemists and biologists may increase the effective gravitational force on a test tube so as to more rapidly and completely cause the precipitate (pellet) to gather on the bottom of the tube. The remaining solution (supernatant) may be discarded with a pipette. There is a correlation between the size and density of a particle and the rate that the particle separates from a heterogeneous mixture, when the only force applied is that of gravity. The larger the size and the larger the density of the particles, the faster they separate from the mixture. By applying a larger effective gravitational force to the mixture, like a centrifuge does, the separation of the particles is accelerated. This is ideal in industry and lab settings because particles that would naturally separate over a long period of time can be separated in much less time.

Principle

Centrifugal force, word from Latin centrum, meaning "center", and fugere, means "to flee", is the apparent force that draws a rotating body away from the center of rotation. It is caused by the inertia of the body as the body's path is continually redirected. Centrifugal force is generated when materials are rotated; the size of the force depends on the radius and speed of rotation and the density of the centrifuged material. The centrifugal force on a particle that is constrained to rotate in a circular path is given by the equation:

F = mω2x (1.1)

Where m is the mass of the particle, ω (omega) is the angular velocity of the spinning rotor in radians per second (one unit of circumference contains 2п radians), and x is the distance of the particle from the axis of rotation.

Where F is the centrifugal force acting on the particle to maintain it in the circular path, r is the radius of the path, m is the mass of the particle, and ω (omega) is the angular velocity of the particle.

Usually, the value given for the force applied to particles during centrifugation is a relative one; that is, it is compared with the force that the earth's gravity would have on the same particles. It is called relative centrifugal f

$$RCF = \frac{F_{centrifugation}}{F_{gravity}} = \frac{m\omega^2 x}{mg} = \frac{\omega^2 x}{g}$$

iven by

(1.2)

Beca

$$\frac{\omega^2}{g} = \frac{\left[\frac{(2\pi)(rpm)}{60}\right]^2}{980}$$

en

(1.3)

In equation 1.3, the term (27r/60)/980 is also a constant and is equal to 1.119 x 10^{-5}.

Thus the equation for relative centrifugal force (i.e., equation 1.2) simplifies to

$$RCF = 1.119 \times 10^{-5} (rpm)^2 \quad (1.4)$$

Consequently, to determine the relative centrifugal force in effect during centrifugation it is necessary to measure the revolutions per minute (rpm) of the rotor and the distance between the sample and the axis of rotation. Because RCF is the ratio of two forces, it has no units. However, it is customary to follow the numerical value of the RCF with the symbol g. This indicates that the applied RCF is a multiple of the earth's gravitational force.

Sedimentation Rate and Sedimentation Coefficient:

The RCF or "g force" applied to particles during centrifugation may readily be calculated using equation 1.4 and is independent of the physical properties of the particles being sedimented. However, a particle's sedimentation rate at a specified RCF depends on the properties of the particle itself.

Also, because the RCF varies directly with x, it is clear that the sedimentation rate changes with changing distance from the axis of rotation. (For particles settling under the influence of the earth's gravity alone, the sedimentation rate is constant.)

The instantaneous sedimentation rate of a particle during centrifugation is determined by three forces:

(1) F_C, the centrifugal force,

(2) F_B, the buoyant force of the medium, and

(3) F_F, the frictional resistance to the particle's movement.

Separation principle

In the separation of immiscible liquids, the denser liquid moves to the bowl wall and the lighter liquid is displaced to an inner annulus. (Figure. 1.1).The thickness of the layers is determined by the density of the liquids, the pressure difference across the layers and the speed of rotation. A boundary region between the liquids at a given centrifuge speed forms at a radius r_n where the hydrostatic pressure of the two layers is equal. This is termed the neutral zone and is important in equipment design to determine the position of feed and discharge pipes. It is found using:

$$r^2_n = \rho_A r^2_A - \rho_B r^2_B \ / \ \rho_A - \rho_B$$

Where ρ (kg/m3) = density and r (m) = the radius. The subscripts A and B refer to the dense and light liquid layers respectively. If the purpose is to remove light liquid from a mass of heavier liquid (for example in cream separation from milk), the residence time in the outer layer exceeds that in the inner layer. This is achieved by using a smaller radius of the outer layer (r1 in Fig.1) and hence reducing the radius of the neutral zone. Conversely, if a dense liquid is to be separated from a mass of lighter liquid (for example the removal of water from oils), the radius of the outer layer is increased.

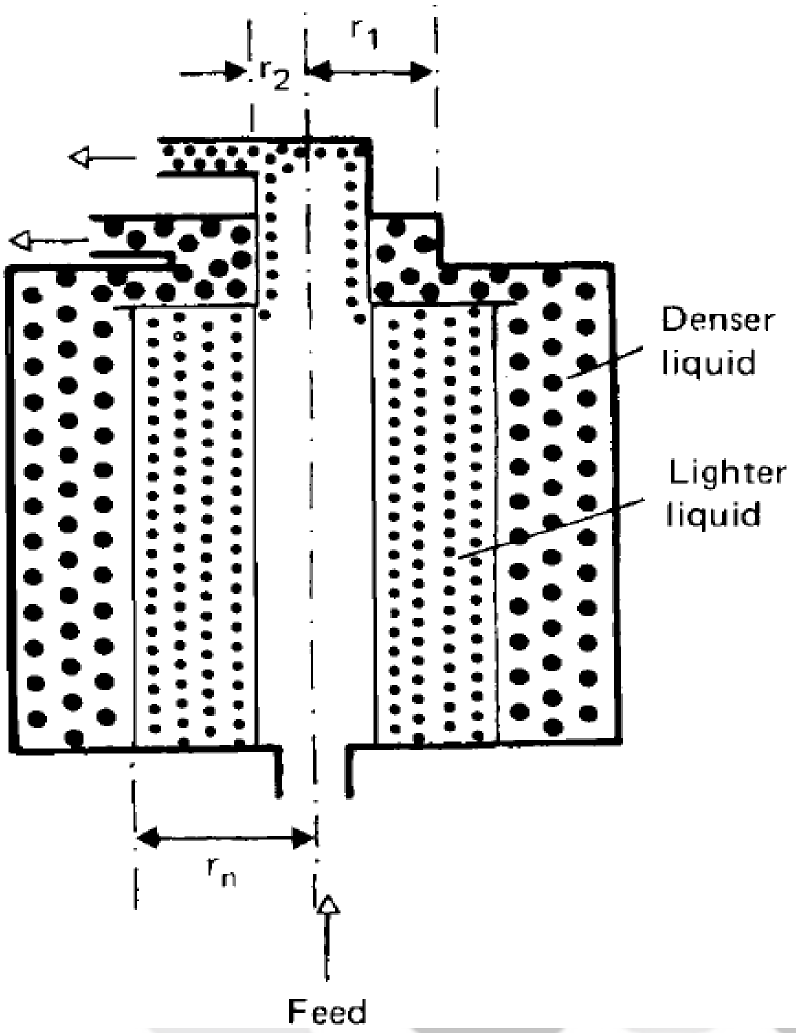

Fig. 1. Separation of immiscible liquids: r1, radius of dense phase outlet; r2, radius of light phase outlet; rn, radius of neutral zone.

Sample problem

A bowl centrifuge is used to break an oil-in-water emulsion. Determine the radius of the neutral zone in order to position the feed pipe correctly. (Assume that the density of the continuous phase is 1000 kg/m3 and the density of the oil is 870 kg /m3. The outlet radii from the centrifuge are 3 cm and 4.5 cm.)

Solution :

$$r_n = \sum \left[\frac{1000(0.045)^2 - 870(0.03)^2}{1000 - 870} \right]$$

$$= \sqrt{\left(\frac{2.025 - 0.783}{130} \right)}$$

$$= 0.097 \text{ m}$$

Rate of Centrifugation

The rate of centrifugation is specified by the angular velocity usually expressed as revolutions per minute (RPM), or acceleration expressed as g. The conversion factor between RPM and g depends on the radius of the centrifuge rotor. The particles' settling velocity in centrifugation is a function of their size and shape, centrifugal acceleration, the volume fraction of solids present, the density difference between the particle and the liquid, and the viscosity. The general formula for calculating the revolutions per minute (RPM) of a centrifuge is

RPM= g/r

where g represents the respective force of the centrifuge and r the radius from the center of the rotor to a point in the sample. However, depending on the centrifuge model used, the respective angle of the rotor and the radius may vary, thus the formula gets modified.

Equipment

A centrifuge is the equipment generally driven by an electric motor that puts an object to rotate around fixed axis, and a perpendicular force is applied to axis. The particles get separated according to their size, shape, density, viscosity of the medium and rotor speed. The separation takes place by subjecting these dispersed systems to artificially induced gravitational fields.

Principle

The centrifuge involves principle of sedimentation, where the acceleration at centripetal force causes denser substances to separate out along the radial direction at the bottom of the tube. By the same concept lighter objects will tend to move to the top of the tube; in the rotating picture, move to the center. In a solution, particles whose density is higher than that of the solvent sink (sediment), and particles that are lighter than it float to the top. The greater the difference in density, the faster they move. If there is no difference in density (isopycnic conditions), the particles stay steady. To take advantage of even tiny differences in density to separate various particles in a solution, gravity can be replaced with the much more powerful "centrifugal force" provided by a centrifuge. Centrifugation can only be used when the dispersed material is denser than the medium in which they are dispersed.

Classification

Centrifuges can be classified on the basis of application and volume handled.

Classification on the basis of application

Centrifuges are classified into three groups for:

1. Separation of immiscible liquids

2. Clarification of liquids by removal of small amounts of solids (centrifugal clarifiers)

3. Removal of solids (desludging or dewatering centrifuges).

Liquid–liquid centrifuges

Tubular bowl centrifuge

The simplest type of equipment is the tubular bowl centrifuge. It consists of a vertical cylinder (or bowl), typically 0.1 m in diameter and 0.75 m long, which rotates inside a stationary casing at between 15000 rev per min and 50 000 rev per min depending on the diameter. Feed liquor is introduced continuously at the base of the bowl and the two liquids are separated and discharged through a circular weir system into stationary outlets (Fig. 1).

Disc bowl centrifuge

Better separation is obtained by the thinner layers of liquid formed in the disc bowl centrifuge (Fig.2). Here a cylindrical bowl, 0.2–1.2 m in diameter, contains a stack of inverted metal cones which have a fixed clearance of 0.5–1.27 mm and rotate at 2000–7000 rev/min. They have matching holes which form flow channels for liquid movement. Feed is introduced at the base of the disc stack and the denser fraction moves towards the wall of the bowl, along the underside of the discs. The lighter fraction is displaced towards the centre along the upper surfaces and both liquid streams are removed continuously by a weir system at the top of the centrifuge in a similar way to the tubular bowl system. Disc bowl centrifuges are used to separate cream from milk and to clarify oils, coffee extracts and juices. Disc bowl and tubular centrifuges have capacities of up to 150 000l/h.

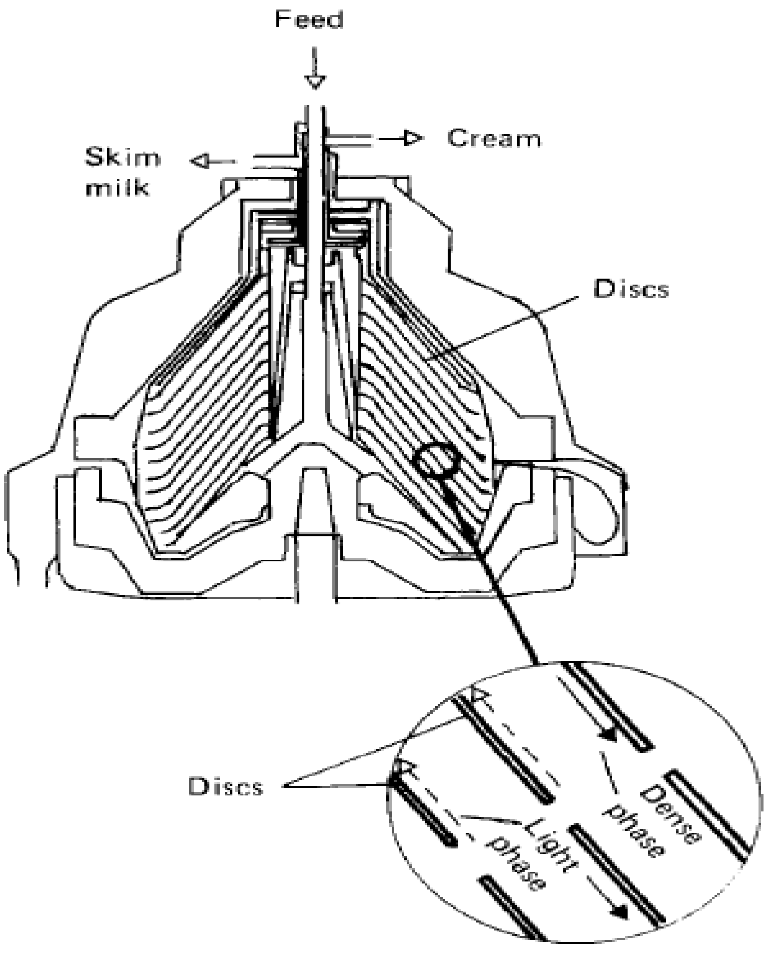

Figure.2. Disc bowl centrifuge.

Centrifugal clarifiers

Solid bowl clarifier

The simplest solid–liquid centrifuge is a solid bowl clarifier, which is a rotating cylindrical bowl, 0.6–1.0 m in diameter. Liquor, with a maximum of 3% w/w solids, is fed into the bowl and the solids form a cake on the bowl wall. When this has reached a pre-determined thickness, the bowl is drained and the cake is removed automatically through an opening in the base.

Nozzle centrifuges or valve discharge centrifuges

Feeds which contain higher solids content (Table 2.) are separated using nozzle centrifuges or valve discharge centrifuges. These are similar to disc bowl types, but the bowls have a biconical shape. In the nozzle type, solids are continuously discharged through small holes at the periphery of the bowl and are collected in a containing vessel. In the valve type the holes are fitted with valves that periodically open for a fraction of a second to discharge the accumulated solids. The advantages of the latter design include less wastage of liquor and the production of drier solids. Both types are able to separate feed liquor into three streams: a light phase, a dense phase and solids. Centrifugal clarifiers are used to treat oils, juices, beer and starches and to recover yeast cells. They have capacities up to 300 000 l h1.

Table. 2. Particle size and solid content handled by centrifuges in food processing.

Centrifuge types	Range of particle sizes (µm)	Solid content of feed (% of w/w)

Disc bowl		
Clarifier	0.5–500	< 5
Self-cleaning	0.5–500	2–10
Nozzle bowl	0.5–500	5–25
Decanter	5–50 000	3–60
Basket	7.5–10 000	5–60
Reciprocating conveyor	100–80 000	20–75

Desludging, decanting or dewatering centrifuges

Feeds with high solids contents (Table 2) are separated using desludging centrifuges, including conveyor bowl, screen conveyor, basket and reciprocating conveyor centrifuges.

Conveyor bowl centrifuge

In the conveyor bowl centrifuge the solid bowl rotates up to 25 rev min1 faster than the screw conveyor (Fig. 3). This causes the solids to be conveyed to one end of the centrifuge, whereas the liquid fraction moves to the other large-diameter end. The solids are relatively dry compared with other types of equipment.

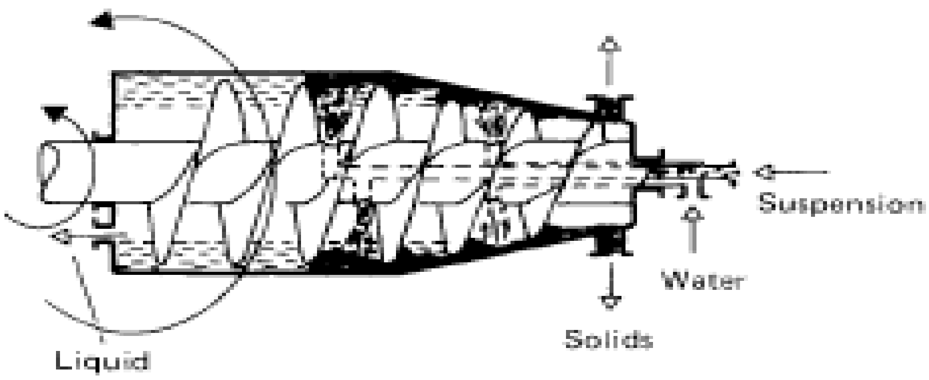

Fig. 6.3 Conveyor bowl centrifuge.
(After Leniger and Beverloo (1975).)

Screen conveyor centrifuge

The screen conveyor centrifuge has a similar design but the bowl is perforated to remove the liquid fraction.

Reciprocating conveyor centrifuge

The reciprocating conveyor centrifuge is used to separate fragile solids (for example crystals from liquor). Feed enters a rotating basket, 0.3–1.2 m in diameter, through a funnel which rotates at the same speed. This gradually accelerates the liquid to the bowl speed and thus minimizes shearing forces. Liquid passes through perforations in the bowl wall. When the layer of cake has built up to 5–7.5 cm, it is pushed forwards a few centimeters by a reciprocating arm. This exposes a fresh area of basket to the feed liquor.

Basket centrifuge

The basket centrifuge has a perforated metal basket lined with a filtering medium, which rotates at up to 2000 rev min1 in automatically controlled cycles which last 5–30 min, depending on the feed material. The feed liquor first enters the slowly rotating bowl; the speed is then increased to separate solids and finally the bowl is slowed and the cake is discharged through the base by a blade. Capacities of these dewatering centrifuges are up to 90 000 l h1 and they are used to recover animal and vegetable proteins, to separate coffee, cocoa and tea slurries and to desludge oils.

Classification on the basis of volume handled

1. Laboratory Centrifuges

2. Preparative Centrifuges

Laboratory Centrifuges

Laboratory centrifuges are used for small-scale separation and clarification (i.e. removal of particles from liquids). Typical liquid volumes handled by such devices are in the range of 1 - 5000 ml. Fig. 4 shows a diagram of a laboratory centrifuge. The material to be centrifuged is distributed into appropriate numbers of centrifuge tubes (which look like test tubes) which are in turn attached in a symmetric manner to a rotating block called the rotor. There are two types of rotors: fixed angle rotors and swing out rotors.

A fixed angle rotor holds the centrifuge in a fixed manner at particular angle to the axis of rotation. Swing out rotors hold the tubes parallel to the axis of rotation while the rotor is stationary but when the rotor is in motion, the tubes swing out such that they are aligned perpendicular to the axis of rotation. When the centrifuge tubes are spun, the centrifugal action creates an induced gravitational field in an outward direction relative to the axis of rotation. and this drives the particles or precipitated matter towards the bottom of the tube. Typical rotation speeds of laboratory centrifuges range from 1,000 - 15,000 rpm. The magnitude of the induced gravitational field is

measured in terms of the G value: a G value of 1000 refers to an induced

field that is thousand time stronger than that due to gravity. The G value which is also referred to as the RCF (relative centrifugal force) value depends on the rotation speed as well as the manner in which the centrifuge tubes are held by the rotor:

$G = r\omega^2/g = 2\pi r n^2/g$

Where

r = distance from the axis of rotation (m)

ω = angular velocity (radians/s)

g = acceleration due to gravity (m/s^2)

n = rotation speed (/s)

Quite clearly the G value in a centrifuge tube will depend on the location, the highest value being at the bottom of the tube and the lowest value being at the top. This implies that a particle will experience increasing G values while moving towards the bottom of the centrifuge tube. In most cases the average G value that a particle is likely to experience i.e. the numerical mean of the maximum and minimum values is used for process calculationsTypical G values employed in laboratory centrifuges range from 1,000 - 20,000.

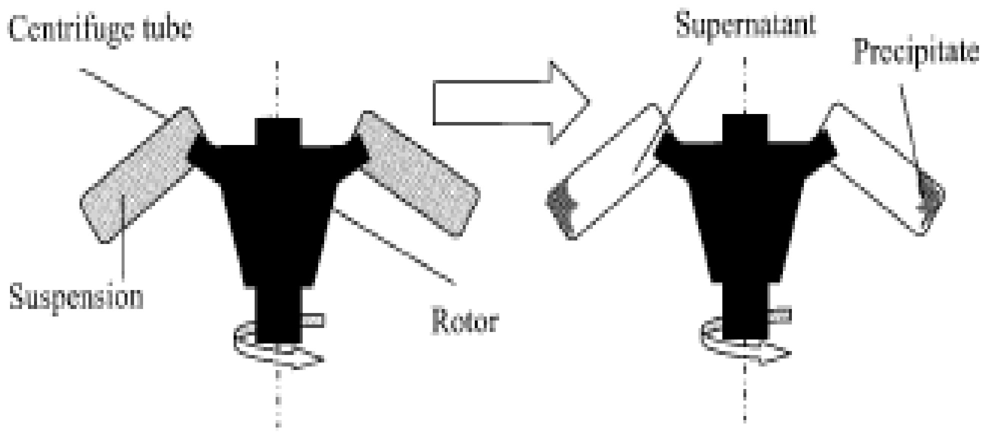

Fig. 4. Laboratory Centrifuge

Preparative centrifuges

Preparative centrifuges can handle significantly larger liquid volumes than laboratory centrifuges, typically ranging from 1 litre to several thousand litres. Preparative centrifuges come in a range of designs, the common feature in these being a tubular rotating chamber. The suspension to be centrifuged is fed into such a device from one end while the supernatant and precipitate are collected from the other end of the device in a continuous or semi-continuous manner. The most common type of preparative centrifuge is the tubular bowl centrifuge as shown in Fig.1. Typical rotating speeds for preparative centrifuges range from 500 - 2000 rpm.

Maintenance of Centrifuge

Quality control

Initial installation

Initial calibration should be performed only by a qualified service technician.