

Centrifugation



Objective

After completion of this topic

- Student will be able to explain Objectives, principle & applications of Centrifugation
- They can explain principles, construction, working, uses, merits and demerits of Perforated basket centrifuge, Non-perforated basket centrifuge, semi continuous centrifuge & Super centrifuge

Introduction

- Centrifugation is an unit operation employed for **separating** the constituents present in a dispersion with the aid of **centrifugal force**.
- Centrifugal force provides **driving force** for separation.
- It replaces gravitational forces operating during **sedimentation**
- It is particularly useful when separation by ordinary filtration is difficult. Like separating highly viscous mixtures and colloidal dispersion (particle size less than 5mm) in which difference in densities is less.
- Centrifugation is convenient method of separating either two immiscible liquids or a solid from a liquid.
- Equipment used for the same is called as **centrifuges**.

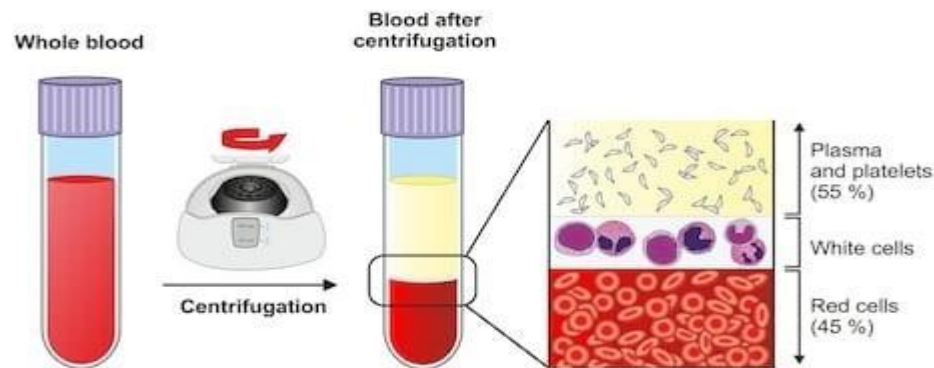


PROCESS OF CENTRIFUGATION:-

- It consist of a container in which a mixture of solid and liquid / two liquids is placed and rotated at high speed.



- The mixture is separated into its constituent parts by the action of centrifugal forces on their densities.
- A solid/ liquid of higher specific gravity is thrown outward with greater force.
- Hence it is retained at the bottom of the container leaving a clear supernatant layer of pure liquid.
- Speed of centrifuge is given in terms of **number of revolutions** per minute of the rotor.



- **APPLICATIONS:-**

1. **Production of bulk products:-** used for separating crystalline drugs such as **aspirin** from the mother liquor. Free flowing products result due to removal of traces from mother liquor and avoidance of effervescence.
2. **Production of biological products:-** most of the proteinaceous drugs and macromolecules are present as colloidal dispersions in water. Hence centrifugal methods are used for separating the same. Insulin is separated from other proteins by precipitation method and then it is finally separated by ultracentrifugation. Also blood products are separated from blood by centrifugation.

- **APPLICATIONS:-**

3. BIOPHARMACEUTICAL ANALYSIS OF DRUGS:- drugs present in blood, tissue fluids and urine are normally present in the form of colloidal dispersions. Centrifugation is used for separating the drugs. This method is easy for evaluation of pharmacokinetic parameters and bioequivalence studies.

- **4. Evaluation of suspension and emulsions:-** it is used for rapid empirical test parameters for the evaluation of suspension and emulsion. Normally creaming is a slow process in emulsion. This process is hastened by inducing stress condition using a centrifuge. A stable emulsion should not show any signs of separation even after centrifuging at 2000-3000 rpm at RT.
- **5. Determination of molecular weight of colloids:-** determination of molecular weight of polymers is not possible by usual methods. Ultracentrifugation methods are used for determination of molecular weight of serum albumin, insulin, methylcellulose.

Principle and theory of centrifugation

- ▶ Centrifugal effect:

Centrifugal force in terms of the ratio to the gravitational force is known as centrifugal effect.

- ▶ Consider a body of mass m rotating in a circular path of radius r at a velocity v . The force acting on the body in a radial direction is given by:

- ▶
$$F = \frac{mv^2}{r}$$

Here, F = centrifugal force

m = mass of the body

v = velocity of body

r = radius of circle of rotation

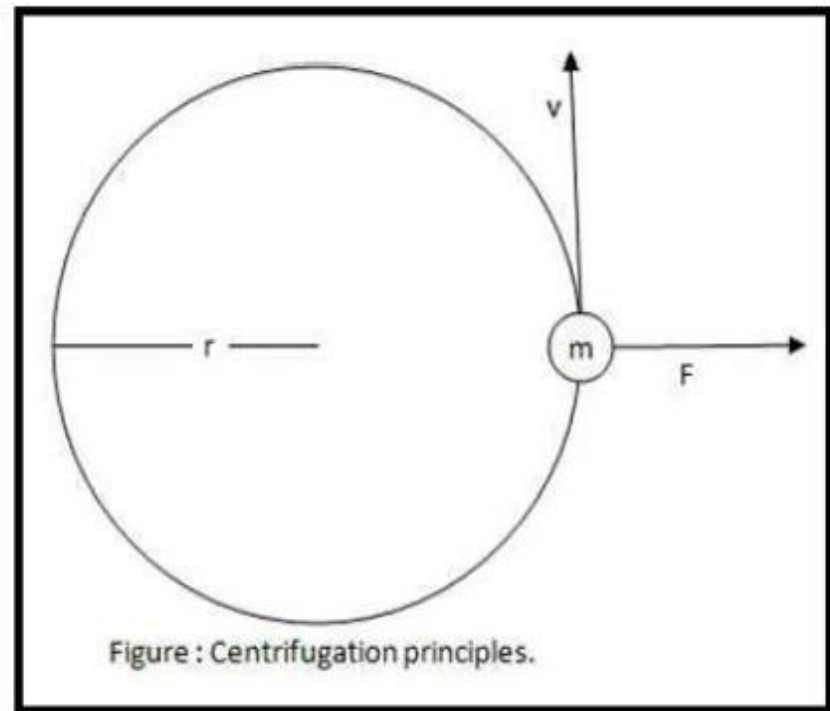


Figure : Centrifugation principles.

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- ▶ The same body will be acted upon by a gravitational force. It can be expressed as:

$$G = mg$$

where, G = gravitational force g = gravitational constant

- ▶ The centrifugal effect is the ratio of the two forces, so that:

$$\begin{aligned} C &= F / G \\ &= mv^2 / mgr \\ &= v^2 / gr \end{aligned}$$

But, $v = 2\pi rn$ Where, n = speed of rotation.

So,

$$\begin{aligned} F / G &= (2\pi rn)^2 / gr \\ &= 4\pi^2 r^2 n^2 / gr \\ &= 2 \pi^2 n^2 d / g \dots\dots\dots(1) \end{aligned}$$

Where, d = diameter of rotation.

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- ▶ The gravitational constant has a value of 9.807 m/s^2 , so that equation (1) may be simplified to:
 - ▶ Centrifugal effect = $2.013 n^2 d$
 - ▶ Provided that , n is expressed in S^{-1} and d is in meter.
 - ▶ From the equation, it can be drawn that the centrifugal effect is directly proportional to the diameter, but is proportional to the square of the speed of the rotation.
 - ▶ Thus, if it is necessary to increase the centrifugal effect, it is of greater advantages to use a centrifuge of the same size at a higher speed, rather than use a
 - ▶ ⁵ larger centrifuge at the same speed of rotation.

Factors affecting centrifugation process

- ▶ Liquid viscosity
- ▶ Liquid density
- ▶ Solids concentration
- ▶ Particle size distribution
- ▶ Surface charge of particles
- ▶ Type and shape of particles
- ▶ Feed rate
- ▶ Agitation speed
- ▶ Size of centrifuge
- ▶ Height of cake
- ▶ Mode of operation
- ▶ Time at full speed

Classification of centrifuge

Industrial centrifuges can be classified into 3 main types:

(1) Filtration centrifuge:

Those using perforated baskets, which perform a filtration type operation (work like a spin dryer).

- ▶ E.g. perforated basket centrifuge, semi-continuous centrifuge

(2) Sedimentation centrifuge:

Those with a solid walled vessel, where particles sediment towards the wall under the influence of the centrifugal force.

- ▶ E.g. Tubular bowl centrifuge, Non-perforated basket centrifuge, super centrifuge, conical disc centrifuge

(3) Continuous centrifuge:

Those where a continuous process or very high capacity is required.

E.g. Continuous Horizontal Centrifuge

Perforated basket centrifuge

- ▶ Principle:
- ✓ Perforated basket centrifuge is a filtration centrifuge. The separation is through a perforated wall based on the difference in the densities of solid and liquid phases.
- ✓ The bowl contains a perforated side-wall. During centrifugation, the liquid phase passes through the perforated wall while solid phase is retained in the bowl.
- ✓ The solid is removed by a blade.
- ✓ In this centrifuge a basket is mounted above a driving shaft such arrangement is described as

under driven

1. Perforated-basket centrifuges (centrifugal filters)

It consists of a stainless steel perforated basket (typically 1-2 m in diameter) lined with a filter cloth.

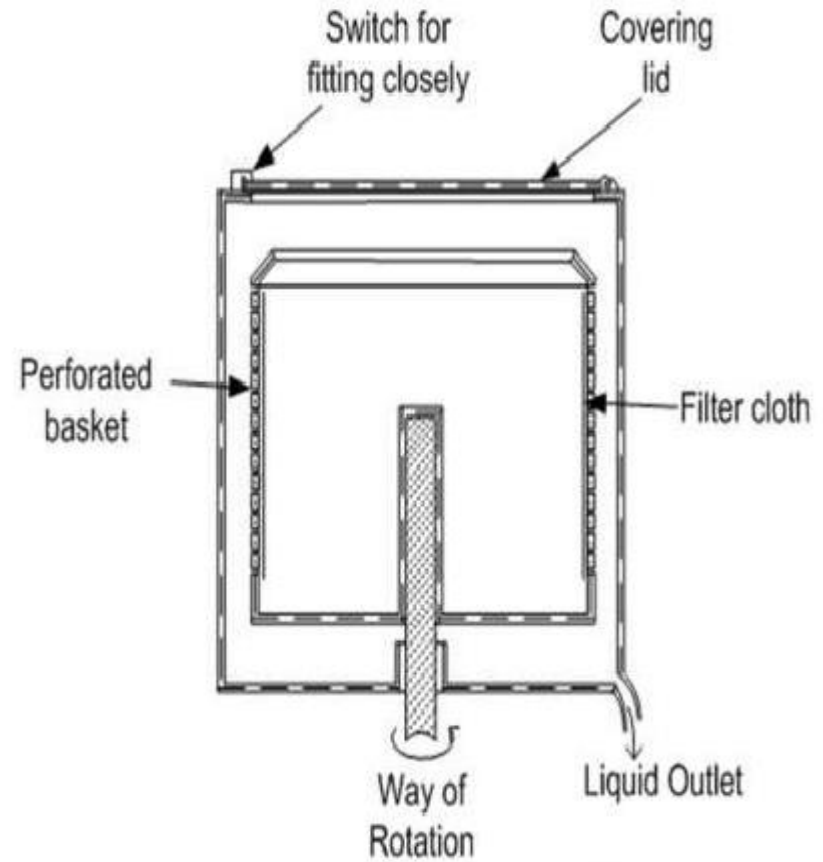
The basket rotates at a speed which is typically $<25 \text{ s}^{-1}$, higher speeds tending to stress the basket excessively.

The product enters centrally and is thrown outwards by centrifugal force and held against the filter cloth.

The filtrate is forced through the cloth and removed via the liquid outlet; the solid material is retained on the cloth.

The cake can be washed if required by spraying water into the centrifuge

Perforated basket centrifuge



Model of perforated basket centrifuge

<http://jeepakistan.blogspot.com>

▶ Working:

- ✓ Liquid feed is inserted to the bowl/basket when it is stationary in such a way that it distributes evenly in the basket.
- ✓ Power is supplied to start the rotation of basket. The basket should achieve its maximum speed quickly. It rotates at 1000 rpm.
- ✓ During the process liquid passes through perforated walls and solid remains inside the wall.
- ✓ The solid is collected after drying by spinning and liquid is collected from outlet.

Uses : (1) Perforated basket centrifuge is extensively used for separating crystalline drugs (such as aspirin) from the mother liquor. Free flowing product can be obtained because mother liquor is removed completely.

(2) It is also used for removing unwanted solids from a liquid. For example, precipitated proteins are removed from insulin.

(3) Sugar crystals are separated using perforated basket centrifuge.

Advantages : (1) The centrifuge is very compact and it occupies very little floor space.

(2) It can handle slurries with a high proportion of solids and even those having paste like consistency.

(3) The final product has very low moisture content.

(4) In this method, the dissolved solids are separated from the cake.

(5) The process is rapid.

Disadvantages : (1) The entire cycle is complicated resulting in considerable labour costs.

(2) It is a batch process.

(3) If the machine is adapted for prolonged operation, there is considerable wear and tear of the equipment. On prolonged operation, the solids may form hard cake, due to the centrifugal force, which is difficult to remove simultaneously.

Semicontinuous centrifuge

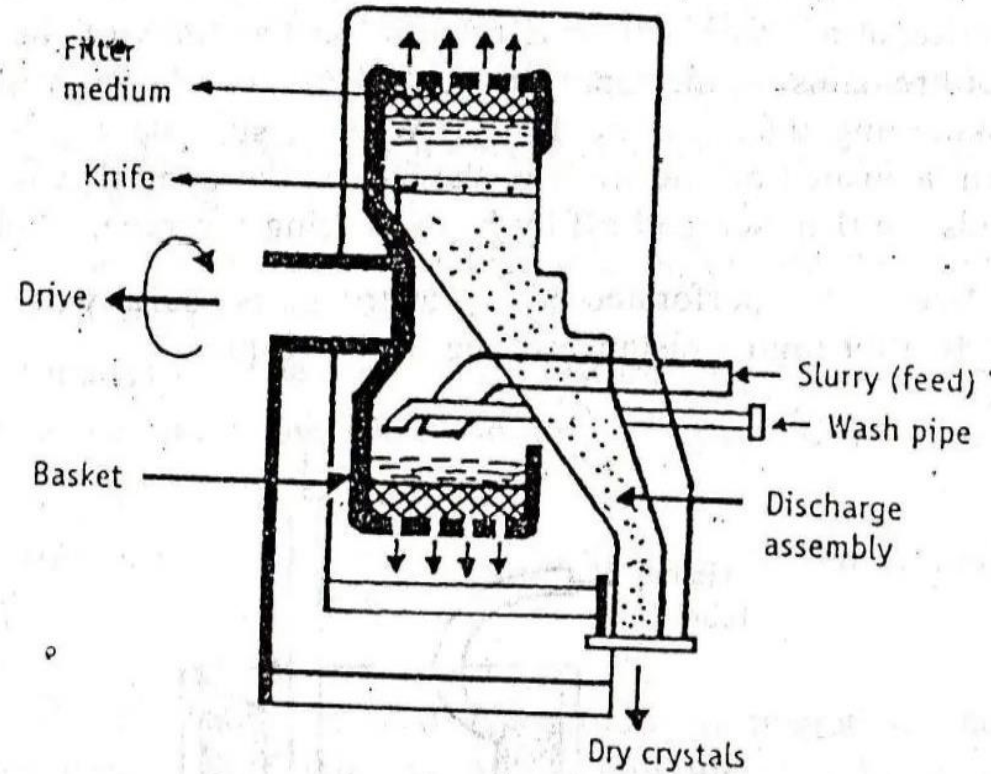


Figure 10-4. Construction of semi-continuous centrifuge.

Semi continuous centrifuge

- ▶ It is also known as short cycle automatic batch centrifuge.
- ▶ Principle:
 - ✓ This centrifuge is a filtration centrifuge. The separation is through a perforated wall based on the difference in the densities of solid and liquid phases.
 - ✓ The bowl contains a perforated side-wall. During centrifugation, the liquid phase passes through the perforated wall while solid phase is retained in the bowl.
 - ✓ The solid is removed by cutting with a blade.

CONSTRUCTION:

- It consist of a vertical perforated basket, which is supported from horizontal shaft driven by motor
- From the open side of the basket, provisions are made at the centre to introduce feed and wash pipe through horizontal tubes.
- A Feeler (not shown in fig.) rides over the feed, which is connected to diaphragm valve through air supply. The feeler controls the thickness of feed.
- Hydraulic cylinder attachment is made in such a manner that the discharge chute enters from the sides of the basket, when discharge of crystals is desirable

▶ Working:

- ✓ The perforated basket is allowed to rotate and Liquid feed is inserted to the bowl/basket from side pipe in such a way that it distributes evenly in the basket.
 - ✓ During the process liquid passes through perforated walls and solid remains inside the wall.
 - ✓ The cake is washed with water. The wash escapes from the basket through the filtrate outlet.
 - ✓ The solid is collected after drying by spinning and liquid is collected from outlet.
 - ✓ In this centrifuge after achieving the desired thickness about 50 to 70 mm the feeler cuts off the air supply to diaphragm valve that automatically cuts of the entry of the slurry.
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ADVANTAGES:

- Short –cycle automatic batch centrifuge is used when solids can be drained fast from the bowl

DISADVANTAGES

- During discharge, considerable breakage of crystals is possible
Many moving parts are involved making construction and functioning more complicated

Super centrifuge

- ▶ Principle:
- ✓ This is sedimentation type centrifuge so separation takes place on the basis of difference in their densities between to immiscible liquids.
- ✓ Centrifugation is done in the bowl of small centrifuge. During centrifugation the heavier liquid is thrown against the wall, while lighter liquid remains as inner layer.
- ✓ The two layers are simultaneously separated using modified wiers.

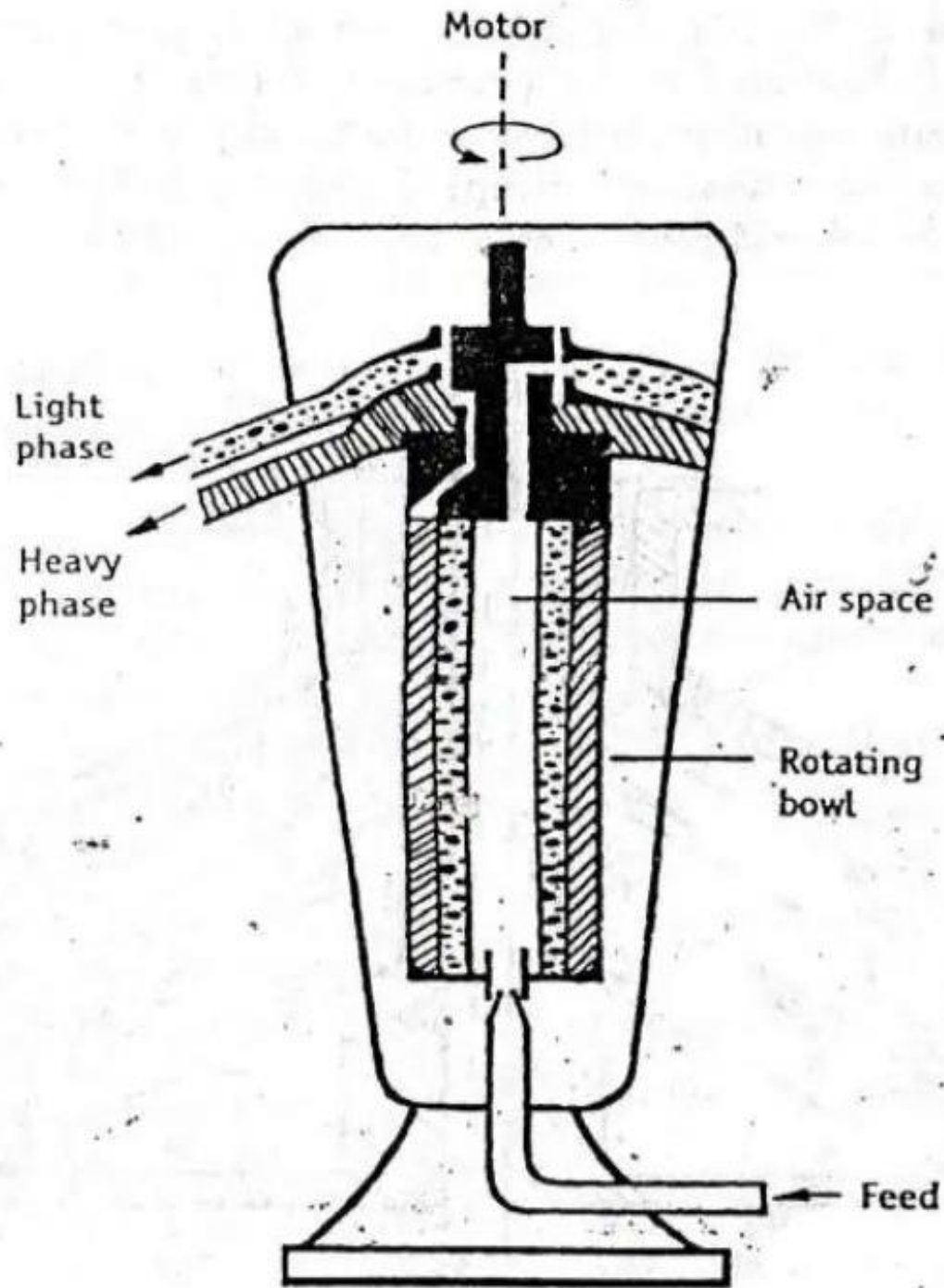
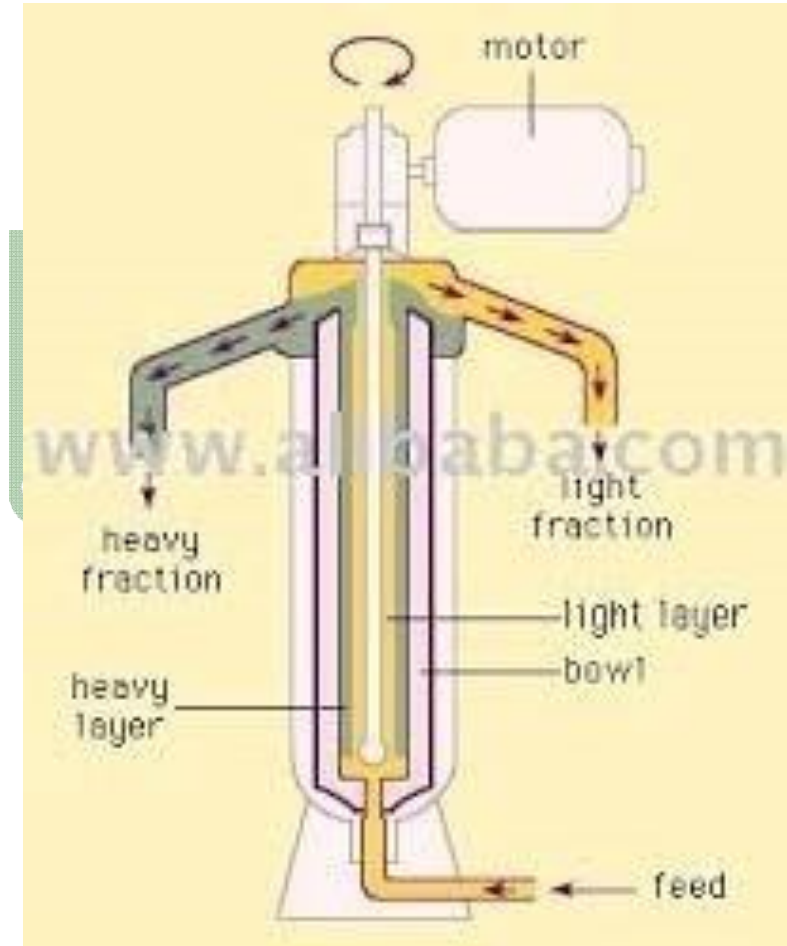


Figure 10-6. Construction of supercentrifuge.

Construction:

- Consist of long hollow cylindrical bowl of small diameter
- It is suspended from a flexible spindle at the top and guided at the bottom by loose-fit bushing
- It can be rotated on its longitudinal axis
- Provision is made at the bottom for the feed inlet using pressure system
- Two liquid outlet are provided at different heights at the top of the bowl and modified weirs are attached



Working

- It is allowed to rotate on its longitudinal axis at a high speed, usually about 2000 revolutions per minutes with the help of drive-assembly
- The feed is introduced from the bottom of the centrifuge using pressure system
- During centrifugation, two liq. Phases separates based on the differences in their densities
- The heavier liquid is thrown against the wall, while the lighter liquid forms an inner layer.
- Both liqs. Rise to the top of the vertical bowl
- The liq-liq interface (neutral zone) is maintained by an hydraulic balance.
- These two layers are simultaneously removed as two liquids separately from different heights through modified weirs
- Thus, the supercentrifuge can work for continuous separation of immiscible liquid phases.

USES

- It is used for separating liquid phases of emulsions in food and pharmaceuticals

Non-perforated basket centrifuge

▶ Principle:

- ✓ This is sedimentation type centrifuge so separation takes place on the basis of difference in their densities without presence of perforated/porous medium.
- ✓ During centrifugation solid remains at side wall of the basket while liquid remains at the top which is collected by skimming tube.

Construction:

- ✓ It consist of basket made up of stainless steel. The basket is placed on the vertical shaft.
 - ✓ Basket contains inlet tube and skimming tube.
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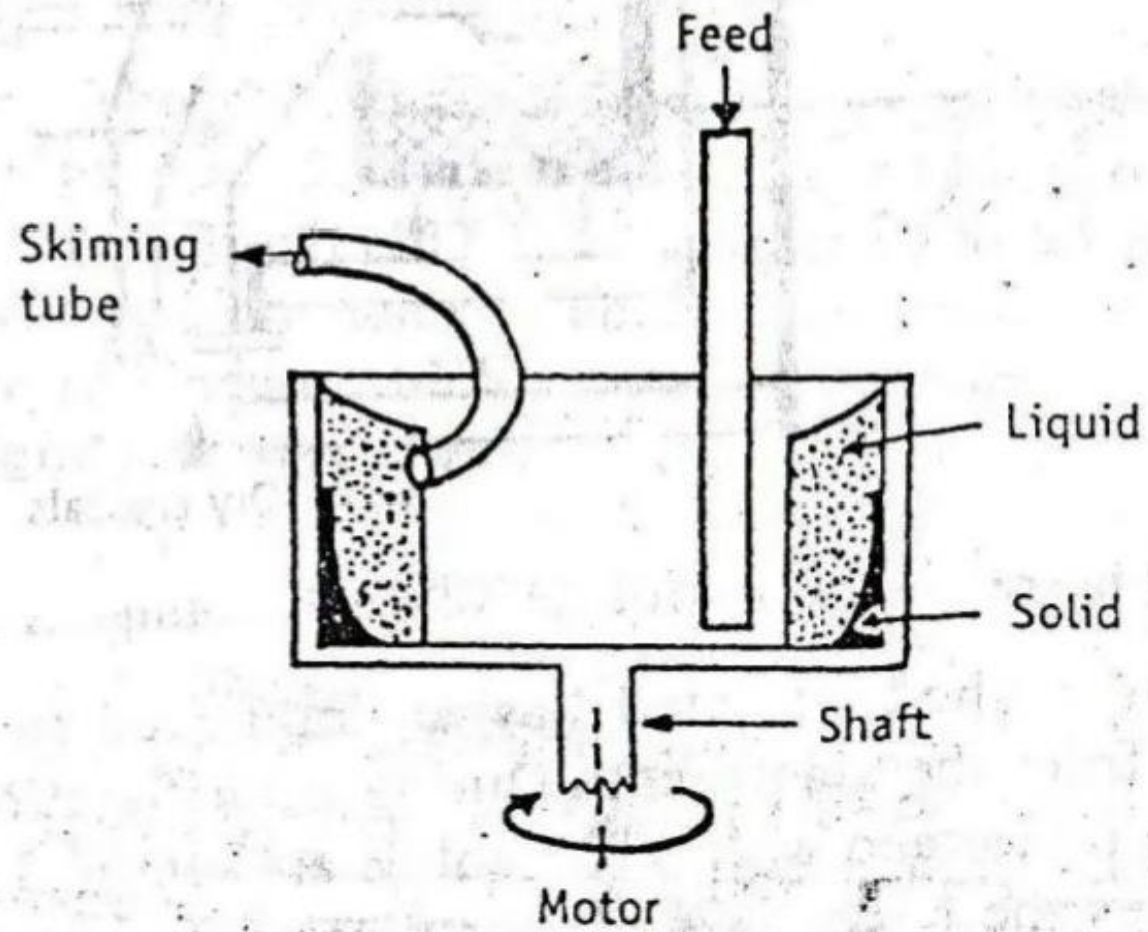


Figure 10-3. Construction of non-perforated basket centrifuge.

▶ Working:

- ✓ The suspension is fed continuously into the basket. During centrifugation solid phase is retained on the sides of the wall while liquid remains on the top.
- ✓ The liquid is collected with skimming tube and solids are scraped off by using scraper from the basket.

▶ Uses:

- ✓ It is useful when the deposited solids offer high resistance to the flow of liquid.

▶ Advantages:

- ✓ Cost is less than other centrifuges

▶ Disadvantages:

- ✓ Solid phase is retained on the sides of the basket.
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THANK YOU