

## REVIEW

**Solid-liquid separations in the food industry: operating aspects and relevant applications**

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**Summary**

Solid-liquid separations in food processing constitute a series of important unit operations that are applied in traditional as well as in innovative processes, and that can play an important role in production, quality and even preservation of many food products. Separation techniques are involved in a great number of processing industries and represent, in many cases, the everyday problem of a practicing engineer. This article reviews general operating principles of solid-liquid separations, under a general fluid mechanics principle, and analyse their main applications in the food industry, emphasizing the basic concept of food quality and preservation.

**Keywords**

particle mechanics; suspension properties; separation efficiency; filtration; centrifugation; hydrocyclone separation; membrane separation

Separation techniques are defined as operations that isolate specific ingredients from a mixture without a chemical reaction taking place. Several criteria have been used to classify or categorize separation techniques. One of such consists in grouping them according to the phases involved, i.e. solid with liquid, solid with solid, liquid with liquid, etc. A classification based on this criterion is shown in Tab. 1. Solid-liquid separation is an important industrial process used for recovery of solids from suspensions and/or purification of liquids in many industries, including the food industry [1]. Solid-liquid separations are widely used in the food industry for a number of tasks, such as concentration and clarification of fruit juices, reduction of microorganisms in fermentation products, separation of coffee and tea slurries, desludging of fish oils, recovering of sugar crystals, treatment of waste water, etc. A basic classification of solid-liquid separations in the food industry is given in Tab. 2. In food processing, some separation techniques such as membrane separations can be used to remove microorganisms and for pasteurization, or even sterilization of liquid foods, without temperature increase. By using physical removal of microorganisms, safety is virtually

guaranteed, while sensory attributes are efficiently preserved.

Finely divided solids in a continuous liquid phase represent common suspensions and slurries in diverse food engineering operations that need to be handled and processed in different manners. A deep understanding of two-phase and multiphase systems is not normally covered in basic courses of study of fluid mechanics and, yet, many flow problems that a food process engineer will face in day to day duties involve some type of non-pure fluid. Slurries and suspensions may behave as non-Newtonian fluids even at lower solids concentrations. Their study may be considered a specific topic of fluid mechanics known as flow through porous media or flow past immersed bodies. It may also be considered a specific topic of rheology, or of particle technology. Simultaneous flow of fluids and solids presents an insight of the main characteristics of suspensions behaviour. Particle-fluid interactions and rheology of suspensions are of the utmost importance for understanding of the phenomena governing relevant solid-liquid separations in food processing industries. Another relevant aspect of solid-liquid separation techniques has to do with effi-

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**Tab. 1.** Classification of separation techniques according to phases involved.

Type of mixture	Technique
Liquid-liquid	Centrifugation Distillation Extraction Decantation Dialysis and electrodialysis Parametric pumping
Solid-solid	Screening Leaching Flotation Air classification
Solid-gas	Cycloning Air filtration Scrubbing Electrostatic precipitation
Solid-liquid	Sedimentation Centrifugation Filtration Membrane separations

ciency of separation and the different manners of expressing it according to the particular technique used.

Mechanical separations involve the movement of solid particles through a fluid, which may be gas or liquid and may be flowing or at rest. The motion of particles through a liquid requires a density difference between the solid particles and the liquid. An external force, normally gravity or centrifugal force, is needed to impart movement to the particle relative to the liquid. The forces acting on a particle moving through a liquid are the gravi-

tational or centrifugal external force, the buoyant force and the drag force. Both the buoyant force and the drag force act parallel to the external force but in the opposite direction. Knowledge of the magnitude of the drag force is essential if the particle motion is to be studied. Conventionally, the drag force  $F_D$  is expressed as:

$$F_D = C_D A \frac{\rho u^2}{2} \quad (1)$$

where  $u$  is the fluid-particle relative velocity,  $\rho$  is the fluid density,  $A$  is the area of the particle projected in the direction of the motion, and  $C_D$  is a coefficient of proportionality known as the drag coefficient.

Assuming that the drag force is due to the inertia of the fluid  $C_D$  will be constant and dimensional analysis shows that  $C_D$  is generally a function of the particle Reynolds number ( $Re_p$ ), i.e.:

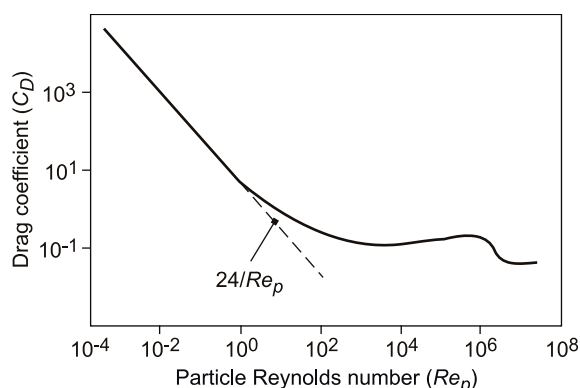
$$Re_p = \frac{ux\rho}{\mu} \quad (2)$$

where  $x$  is the particle size and  $\mu$  is the medium viscosity; the form of the function depends on the flow regime. This relationship for rigid spherical particles is shown in Fig. 1.

For low Reynolds numbers, the drag force for a sphere conforms to a theoretical equation called the Stokes' law, which in theory is valid for values of Reynolds numbers less than about 2, as shown in Fig. 1. Many solid-liquid separations of industrial interest are concerned with fine particles, which are the most difficult to separate, so the Reynolds numbers are low and it is reasonable to

**Tab. 2.** Solid-liquid separation techniques relevant in food processing.

Type of separation	Technique	Examples
Insoluble solids from liquids	Sedimentation	Waste water treatment; separation of sugar crystals from mother liquor; cleaning of incoming raw materials
	Centrifugation	Clarification of juices and beverages; dewatering of vegetable oils; desludging of animal oils
	Hydrocyclone separation	Corn and potato starch refining; cottonseed oil processing; extraction of soluble coffee; clarification of fruit juices
	Filtration	Sugar extraction; dewatering of starch; separation of gluten suspensions; refining of edible oils; clarification of juices and beverages
Soluble solids from liquids	Membrane separations	Desalting of brine; recovery of lactose from whey; sugar recovery in confectionery; clarification and sterilization of fruit juices; brewing products
	Crystallization	Refining of sugar from sugarcane and sugar beet; salt manufacture; winterization of oil
Liquids from solids	Pressing	Extraction of oil from seeds; removal of crude juice from fruits; expressing juices from sugarcane
	Drying	Heated-air dehydration of fruits and vegetables; drying of meat and meat products; fluidized-bed drying of grains



**Fig. 1.** Drag coefficient versus particle Reynolds number for spherical particles.

consider only the Stokes' region. A common expression of the Stokes' law is:

$$u_t = \frac{x^2(\rho_s - \rho)g}{18\mu} \quad (3)$$

where  $\rho_s$  is the particle density,  $g$  is the acceleration due to gravity, and  $u_t$  is known as the terminal settling velocity under gravity.

If a suspension is subjected to a centrifugal force to aid separation of fine particles, the inclusion of the centrifugal term in Eq. (3) leads simply to:

$$v_r = \frac{x^2(\rho_s - \rho)R\omega^2}{18\mu} \quad (4)$$

where  $v_r$  is the radial settling velocity,  $R$  is the ra-

dius of the particle position and  $\omega$  is the angular velocity.

Another situation of fluid-particle relative velocity is that in which fluids flow through beds of solid particles, such as in filtration and in the two-phase counter-current flow of liquid and gas through packed towers. In filtration, the solids bed is constituted by small particles being removed from the liquid by a porous medium. The resistance to the flow of a fluid through the voids in a solids bed is the resultant of the drag of all the particles in the bed. Calculations of the total pressure drop through a bed of solids can be based on estimates of the total drag of the fluid on the solid boundaries of the tortuous channels through the bed of particles [2]. The total drag per unit area of channel wall would be the sum of viscous drag forces and inertial forces, while a direct determination of the total surface area of the bed would be the sum of the individual surface areas of all particles within the bed. Since this latter approach would be unpractical and unattainable, total surface area could be derived from estimation of the relation surface-volume for the solids in the bed.

Flow of suspensions is normally studied by rheology and, in the most general sense, any fluid response not explainable by Newtonian theory may be termed non-Newtonian. The rheological properties of suspensions have been studied since the beginning of last century. The first research was done by EINSTEIN [3, 4] in his classical study of the viscosity of dilute suspension of rigid spheres. The effect of concentration is normally contemplated in models such as Einstein's, and several correlations to correct the viscosity of a suspension are available.

**Tab. 3.** Classification of fluids.

Fluids	Characteristic function	Examples
I. Newtonian	$\tau = \mu\dot{\gamma}$	Air; water; steam; all gases; most fluids consisting of simple molecules
II. Non-Newtonian		
1. Time independent		
a) Herschel-Bulkley	$\tau = \tau_0 + K\dot{\gamma}^n$	Drilling fluids; concrete mixtures
b) Shear thickening (dilatant)	$\tau = K\dot{\gamma}^n; n > 1$	Quick sand; thick starch solutions; wet beach sand; fine powders in suspension
c) Shear thinning (pseudoplastic)	$\tau = K\dot{\gamma}^n; n < 1$	Paper pulp; paint
2. Time dependent non-elastic		
a) Rheopectic	No unique	Bentonite clay
b) Thixotropic	No unique	Paints; printing inks; tomato ketchup; oil drilling mud
3. Viscoelastics	No unique	Saliva; nearly all biological fluids; concentrated tomato soup; bread dough; many polymeric solutions

A general classification of fluids is presented in Tab. 3. A wide variety of non-linear relationships between stress and shear rate have been used. Possibly the most common one is that known as the power law. Fluids described by the power law are generally known as power law fluids. A plot of shear stress against shear rate for power law fluids in logarithmic scale will give straight lines with slope  $n$  and intercept on the ordinate  $K$ . If the slope of such an obtained line is less than one, the material flows more easily the faster it is sheared, and the apparent viscosity decreases with an increasing shear rate. These fluids are known as shear-thinning or pseudoplastic fluids. On the other hand, if the slope is greater than one, the apparent viscosity increases with the increasing shear rate. Such fluids are termed shear-thickening or dilatant fluids. The intercept  $K$  is termed the fluid consistency index, while the slope  $n$  is known as the flow behaviour index. Both constants are used to characterize non-Newtonian fluids.

The solid phase of suspensions to be treated in solid-liquid separation equipment generally consists of an immense number of particles of diverse sizes and shapes. All this population of particles needs to be identified or characterized. The frequency of occurrence of particles of every size present, arranged and presented in a statistical manner, is known as the particle size distribution [5]. In solid-liquid separation, it is important to know the particle size distribution in order to identify which part of it will be separated, and transform this quantity to a measure of efficiency. Normally, particle size data for solid-liquid separation purposes are presented as a plot. What is plotted on the particle size axis is a matter of which quantity used to represent the size of indi-

vidual particles suits better the specific problem. A number of quantities, such as the equivalent sphere diameter, have been used for representing the particle size [6]. The equivalent sphere is the diameter of a sphere which has the same property as the particle itself. Such a property could be the settling velocity. A diameter derived from the settling velocity is known as the Stokes' diameter and is a very useful value for solid-liquid separations, in particular to those techniques in which the particle motion relative to the fluid is the governing mechanism. In general, particle size distribution can be presented as frequencies  $f(x)$  or cumulative frequencies  $F(x)$ , which are related to each other by following relation:

$$f(x) = \frac{dF(x)}{dx} \quad (5)$$

The graphical representation of a particle size distribution can be plotted in cumulative form in which points are entered showing the amount of particulate material contributed by particles below or above a given size. Oversize and undersize distributions are related by (Fig. 2):

$$F(x)_{\text{oversize}} = 1 - F(x) \quad (6)$$

where  $F(x)$  is the cumulative fraction undersize.

A cumulative plot will include a broad range of particle sizes but it is often convenient, however, to refer to a single characteristic size for the system. An important value, which can be read from any cumulative plot of the particle size data, is the median particle size. It is defined as the particle size for which the particle amount equals 50% of the total. A single efficiency number can never facilitate full description of the result of separation,

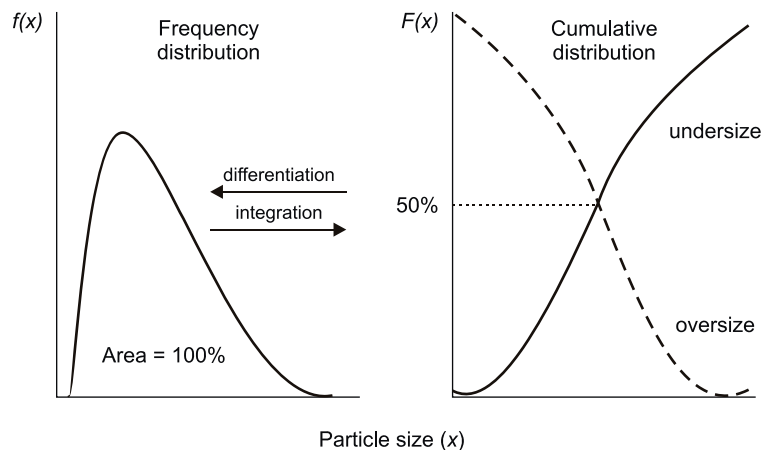
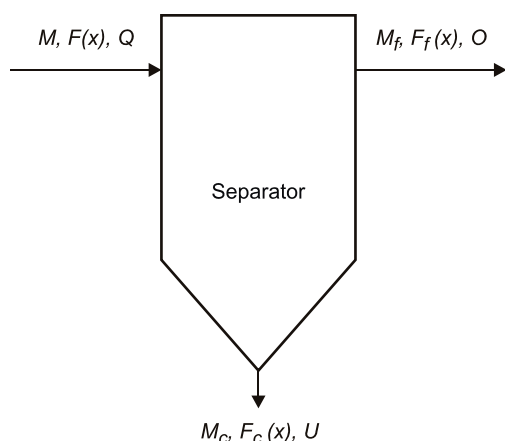


Fig. 2. Relationship between frequency and cumulative particle size distributions.



**Fig. 3.** Schematic diagram of a separator.

$M$  – mass flow rate of solids in the feed,  $M_c$  – mass flow rate of separated solids,  $M_f$  – mass flow rate of non-separated solids,  $F(x)$  – cumulative percentage oversize of feed solids,  $F_c(x)$  – cumulative percentage oversize of separated solids,  $F_f(x)$  – cumulative percentage oversize of non-separated solids,  $Q$  – volumetric flow rate of feed suspension,  $U$  – volumetric flow rate of underflow suspension,  $O$  – volumetric flow rate of overflow suspension.

except when it is ideal. The imperfection of solid-liquid separation has caused the need to express the efficiency by different means. The first and most obvious definition of separation efficiency is simply the overall mass recovery as a fraction of the feed flow rate, according to Fig. 3:

$$E_t = \frac{M_c}{M} \quad (7)$$

where all the components are as defined in the mentioned figure.

If there is no accumulation of solids in the separator then:

$$M = M_c + M_f \quad (8)$$

and there is a choice of three possible combinations of the material streams for the total efficiency testing. It can be shown [7] that if all the operating conditions are identical, the most accurate estimation of the local efficiency comes from the two leaving streams.

The total efficiency defined by Eq. (7) includes all particle sizes present in the feed solids. If only a narrow range of particle sizes is of interest, another efficiency of separation particular to that range can be defined. A mathematical expression of such partial efficiency is:

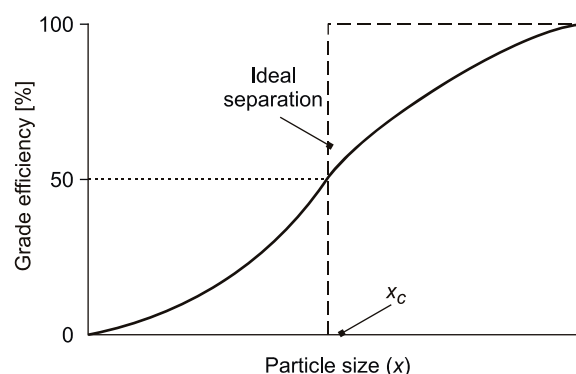
$$E_p = \left( \frac{M_c}{M} \right)_{x_1/x_2} \quad (9)$$

where  $x_1$  and  $x_2$  represent the particle size limits of a definite range.

If the particle size range in Eq. (9) becomes infinitesimal, the obtained efficiency corresponds to a single particle size  $x$  and it is known as the grade efficiency, which is defined by:

$$G(x) = \left( \frac{M_c}{M} \right)_x \quad (10)$$

The grade efficiency of most separation equipments is a continuous function of  $x$ . An S-shaped curve is usually obtained for separators in which inertial or gravity body forces perform the separation. Plotting the probability for any given size fraction against particle size would give a curve as shown in Fig. 4. This sort of curve is normally called a grade efficiency curve, in which the size at the 50% value will represent the limiting size of fine and coarse fraction of a particle size distribution, and is useful to define separation criteria in dynamic separators. Such a size, used to interpret efficiency, is known as the cut size or cut point in separators like centrifuges and hydrocyclones.



**Fig. 4.** Grade efficiency curve for dynamic separators.

## CENTRIFUGATION

Centrifugation is a unit operation involving the separation of materials by the application of centrifugal force. In solid-liquid separations, typical applications of centrifugation are centrifugal clarification, slurry centrifugation and centrifugal filtration. Centrifugal clarification is the term used to describe the removal of small quantities of insoluble solids from a fluid by centrifugal means. If a dilute suspension containing solids with a greater density than the liquid is fed to a rotating cylindrical bowl, the solids will move towards the bowl wall. If an outlet is provided for the liquid



near the centre of rotation, then those particles of solid which reach the bowl wall will remain in the bowl. Those particles which do not reach the bowl wall will be carried out in the liquid. The fraction remaining in the bowl and the fraction passing out in the liquid will be controlled by the feed rate. If a solid particle of diameter  $x$  moves radially in a liquid within a rotating bowl at its terminal velocity under laminar flow conditions, the radial velocity of the particle will be represented by Eq. (4). The time required for a particle to travel an elemental radial distance,  $dR$ , is:

$$dt = \frac{dR}{v_r} = \frac{18\mu}{\omega^2(\rho_s - \rho)x_c^2} \cdot \frac{dR}{R} \quad (11)$$

Assuming that half of all those particles present in the feed with a particular diameter,  $x_c$ , are removed during their transit through the bowl, those particles with diameters greater than  $x_c$  will be mostly removed from the liquid, whereas those particles with diameters smaller than  $x_c$  will be likely to remain in the liquid. The size  $x_c$  is known as the “cut-point” or “critical” diameter.

If clarification is taking place in a simple cylindrical centrifuge, all particles of diameter  $x_c$  contained in the outer half of the cross sectional area of the ring of liquid will reach the bowl wall and will be removed from the liquid. The maximum distance that a particle in this zone has to travel to reach the bowl wall can be represented by the relation of radii:  $R_2 - [(R_1^2 + R_2^2)/2]^{1/2}$ , where  $R_2$  is the radius of the bowl and  $R_1$  is the radius of the annular space of air in the bowl. The time required for a particle of diameter  $x_c$  to travel this distance is:

$$t = \frac{18\mu}{\omega^2(\rho_s - \rho)x_c^2} \int_{[(R_1^2 + R_2^2)/2]^{1/2}}^{R_2} \frac{dR}{R} \quad (12)$$

Integrating Eq. (12) and substituting limits, the following relation is obtained:

$$t = \frac{18\mu \ln \left( \frac{R_2}{[(R_1^2 + R_2^2)/2]^{1/2}} \right)}{\omega^2(\rho_s - \rho)x_c^2} \quad (13)$$

The minimum residence time for a particle in the bowl is  $V/Q$ , where  $V$  is the volume of liquid held in the bowl at any time and  $Q$  is the volumetric flow rate of liquid through the bowl. Thus, for a particle of diameter  $x_c$  to be separated out:

$$\frac{V}{Q} = \frac{18\mu \ln \left( \frac{R_2}{[(R_1^2 + R_2^2)/2]^{1/2}} \right)}{\omega^2(\rho_s - \rho)x_c^2} \quad (14)$$

Eq. (14) may be written in the form:

$$Q = 2 \left[ \frac{g(\rho_s - \rho)x_c^2}{18\mu} \right] \cdot \left[ \frac{\omega^2 V}{2g \ln \left( \frac{R_2}{[(R_1^2 + R_2^2)/2]^{1/2}} \right)} \right] \quad (15)$$

Another way of expressing Eq. (15) is as follows:

$$Q = 2u_g \Sigma \quad (16)$$

where  $u_g$  is the terminal settling velocity of a particle of diameter  $x_c$  in a gravitational field and  $\Sigma$  is a characteristic parameter of any given centrifuge equivalent to the area of a gravity settling tank with settling characteristics similar to the centrifuge. Different values of  $\Sigma$  are given in the literature [8]. For a simple cylindrical bowl centrifuge:

$$\Sigma \approx \frac{\pi \omega^2 b (3R_2^2 + R_1^2)}{2g} \quad (17)$$

where  $b$  is the height of the bowl. Also, for a disc-bowl centrifuge:

$$\Sigma = \frac{2\pi \omega^2 (S-1)(R_x^3 - R_y^3)}{3g \tan \Omega} \quad (18)$$

where  $S$  is the number of discs in stack,  $R_x$  and  $R_y$  are the outer and inner radii of stack, respectively, and  $\Omega$  is the conical half angle of discs [9].

Tubular-bowl machines and disc-bowl separators have found diverse application in the food processing industry. Some important examples include dewatering of vegetable and fish oils, clarification of sweet juices and fermented products, separating of cream from milk, recovery of yeasts, dewatering of different starches, etc. In treatment of fruit juices, beer and wines, the quality of the clarified products may be directly related to the centrifugation efficiency in terms of removing protein fractions to avoid undesirable reactions such as browning. SAPERS [10] investigated filtration and centrifugation as means of preventing enzymatic browning in minimally processed fruit juices. Centrifugation prevented browning in pear juice, as well as in Granny Smith, Golden Delicious and Red Delicious apple juices. Browning was not prevented, however, in McIntosh apple juice. Centrifugation has also been used solely as the clarification step in fruit juice processing, like a partially clarified apple juice that can be obtained by treat-

ing the pressed, screened juice by centrifugation only [11].

In some other applications, for example in starch refining, the use of “sour liquid” (liquid fermentate from traditional fermentation processes) aids sedimentation and competes with centrifugation in the quality of the obtained product. Mung bean starch produced by sour liquid processing had better quality than that from centrifugation, which is the technique commonly used in China to produce starch noodles [12]. The structures of mung bean starch from the two different processing methods were studied and it was shown that light transmissivity of mung bean starch from sour liquid processing was higher than that from centrifugation. The size of mung bean starch from sour liquid processing was as large as that from centrifugation. It has been also reported on properties and qualities of vermicelli made using these techniques [13]. Vermicelli made from sour liquid starch had more significant mesh structure than vermicelli made by centrifugation, while total cooking loss value of vermicelli made from sour liquid starch was significantly lower. Soluble saccharides and proteins were recovered from yam tubers [14] using two consecutive centrifugations at a rotational speed of  $7300 \times g$ . Total amounts of 4.8% of saccharides and 33.8% of proteins were recovered from Keelung yam (*Dioscorea pseudojaponica* Y.). These percentages were increased using foam fractionation to 98.8% and 74.1%, respectively. Continuous-flow

centrifugation has been successfully used for concentration and separation of bacterial spores contaminating whole milk and skimmed milk [15]. *Bacillus subtilis*, *Bacillus atrophaeus*, and *Clostridium sporogenes* spores were recovered from samples of milk at 167 Hz and  $0.7 \text{ l} \cdot \text{min}^{-1}$  flow rate. Spore recoveries ranged from 55% to 88.2%, so continuous flow centrifugation can be suggested as a quick method of sanitizing milk. STREIT et al. [16] reported about the effect of centrifugation conditions on the quality of *Lactobacillus bulgaricus* CFL1 starters. Centrifugation conditions did not significantly affect the acidification activity, while speed and duration of centrifugation slightly affected the cell resistance to freezing. The cryotolerance was improved combining good centrifugation conditions and acidification of the cells in their fermented broth.

## HYDROCYCLONE SEPARATION

The use of hydrocyclones is another possibility to separate suspended solids from a liquid taking advantage of the centrifugal force. As shown in Fig. 5, a hydrocyclone consists of a cono-cylindrical body, which promotes vortex formation when a suspension is pumped through it. The vortex creates a centrifugal force, which causes coarse particles to migrate against the cyclone wall and be discharged by the underflow orifice. Fine particles remain around the central axis of the cyclone and

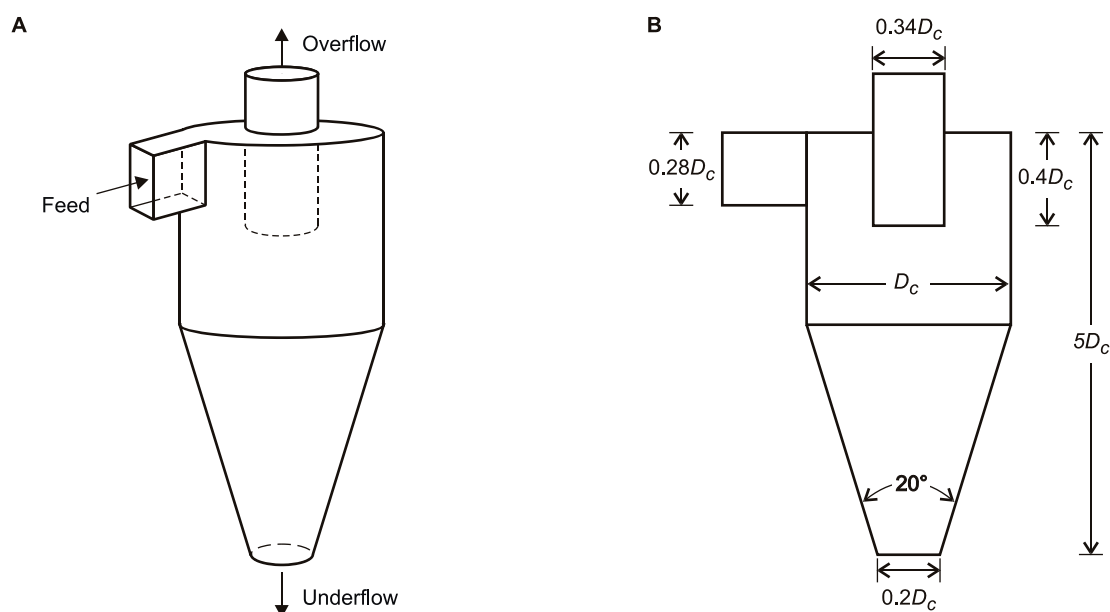


Fig. 5. Description of a hydrocyclone.

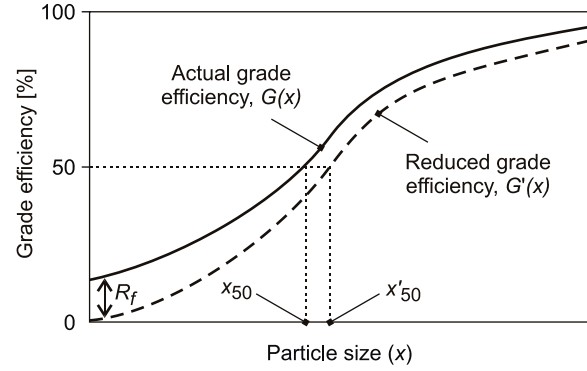
A – Flow diagram, B – Rietema's standard geometry.

are carried out by the overflow stream. Hydrocyclones are easily manufactured and modified, and have been well tested in thickening, clarification, classification and other operations in many industries.

Similarly to centrifuges, hydrocyclones may be evaluated in terms of separation efficiency by means of the cut size or cut point ( $x_c$  or  $x_{50}$ ). The cut size, which is the only single number that in some way represents the separating capability of a hydrocyclone, is the particle size at which the grade efficiency  $G(x)$  curve shows a value of 50%. A grade efficiency curve for a hydrocyclone is derived from screen analysis data on the feed, overflow and underflow streams, and is a continuous representation of the overall mass recovery as a fraction of the mass flow rate. Since most suspended powders and fine particulate systems can be represented by a continuous size distribution, the grade efficiency curve is really derived from a step-wise calculation, drawing a line through the mid-points of size intervals [7]. The grade efficiency curve is an S-shaped cumulative plot in which the 50% point represents a limit value. ORTEGA-RIVAS [17] reviewed some of the numerous expressions utilized for determining the cut size.

The grade efficiency curve gives a plot that does not pass through the origin. This can be explained considering that a hydrocyclone is a flow divider, so the underflow always contains a certain quantity of very fine particles which simply follow the flow, and are split in the same ratio as the liquid. The apparent finite efficiency for fine particles is therefore equal to the underflow-to-throughput ratio  $R_f$ , and a “corrected” or “reduced” cut size  $x'_{50}$  will practically assess the performance of hydrocyclones when derived from the reduced grade efficiency  $G'(x)$ . All these definitions are given in Fig. 6.

Since hydrocyclones have no rotating parts and the vortex action to produce centrifugal force is obtained by pumping the feed suspension tangentially into the cono-cylindrical body, the literature is full of studies on the effects of relative geometric proportions on pressure drop or capacity and separation efficiency. Using this information, any given hydrocyclone geometry could be selected to obtain an optimum performance in terms of cut size. In this sense, possibly the best way to predict hydrocyclone performance is the use of a dimensionless scale-up model well-described elsewhere [18]. Three dimensionless groups can be used to describe hydrocyclone operation and performance: the Euler number  $Eu$ , the Reynolds number  $Re$  and the Stokes number  $Stk_{50}$ . For best application of the relationships among dimensionless



**Fig. 6.** Grade efficiency and reduced grade efficiency curve for hydrocyclone.

groups, certain proportions must be unchanged. Such proportions are generally reported as a function of the diameter of the hydrocyclone. There are several different standard hydrocyclone designs in which proportions remain the same regardless of the size. One of the most efficient designs for separation is called the Rietema cyclone [19], whose proportions are illustrated in Fig. 5. Some other standard hydrocyclones are known as the Bradley's design, the Mozley (1, 2 and 3) cyclone, the Warman 3" model R, and the RW 2515 (AKW) unit [20].

The Euler number, which is a pressure loss factor, is defined as the limit of the maximum characteristic velocity  $v$  obtained by a certain pressure drop  $\Delta P$  across the cyclone as:

$$Eu = \frac{2\Delta P}{\rho v^2} \quad (19)$$

where  $\rho$  is the liquid density and  $v$  is the superficial velocity in the cyclone body.

The Reynolds number defines flow features of the system and, in the case of hydrocyclones, the characteristic dimension may be the cyclone body diameter  $D_c$ :

$$Re = \frac{D_c v \rho}{\mu} \quad (20)$$

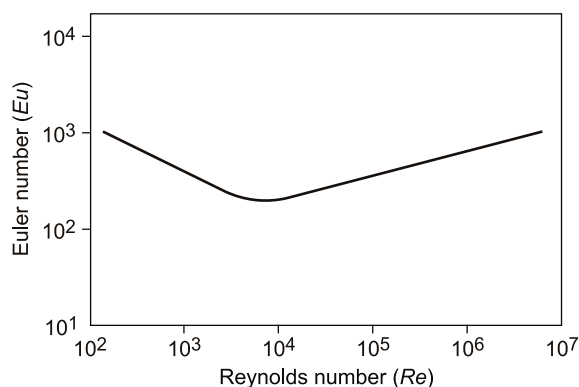
where  $\rho$  is the liquid density and  $\mu$  is the average viscosity.

The Stokes number may be derived from basic fluid mechanics theory and is defined as follows:

$$Stk_{50} = \frac{x_{50}^2 (\rho_s - \rho) v}{18 \mu D_c} \quad (21)$$

where  $x_{50}$  is the cut size,  $\rho_s$  is the solids density and  $D_c$  is the hydrocyclone diameter.





**Fig. 7.** A typical plot of  $Eu$  versus  $Re$  for hydrocyclones.

All the above equations use the superficial velocity in the cyclone body as the characteristic one, i.e.:

$$v = \frac{4Q}{\pi D_c^2} \quad (22)$$

where  $Q$  is the feed volumetric flow rate.

The dimensional analysis gives two basic relationships between the dimensionless groups mentioned above:

$$Stk_{50} Eu = \text{constant} \quad (23)$$

$$Eu = k_p (Re)^{n_p} \quad (24)$$

where  $k_p$  and  $n_p$  are constants derived for a family of geometrically similar hydrocyclones.

These relationships have been tested over a range of conditions by different workers [21–23]. Their plots are typical, as shown in Fig. 7 for Eq. (24).

At higher concentrations, the feed concentration as a fraction of volume  $C$  has to be included as an additional dimensionless group. SVAROVSKY and MARASINGHE [24] reported the following expression for the effect of high concentrations of solids in the feed:

$$Stk_{50}(r) = k_1(1 - R_f)e^{k_2 C} \quad (25)$$

where  $Stk_{50}(r)$  includes the previously described reduced cut size, while  $R_f$  is the underflow-to-throughput ratio.

An exhaustive study for concentrations up to 10% by volume was carried out by MEDRONHO and SVAROVSKY [25] to verify the applicability of Eqs. (23), (24), and (25). Using three geometrically similar hydrocyclones of Rietema's optimum geometry they obtained:

$$Stk_{50}(r)Eu = 0.047 \left( \ln \frac{1}{R_f} \right)^{0.74} e^{(8.96C)} \quad (26)$$

$$Eu = 71 (Re)^{-0.116} \left( \frac{D_i}{D_c} \right)^{-1.3} e^{(2.12C)} \quad (27)$$

$$R_f = 1218 \left( \frac{D_u}{D_c} \right)^{-4.75} (Eu)^{-0.30} \quad (28)$$

where  $D_i$ ,  $D_c$ , and  $D_u$  are the inlet, body and underflow diameters of the hydrocyclone, respectively.

For concentrations higher than 10% by volume, many practical slurries show non-Newtonian behaviour and it can be shown [18] that expressions including Reynolds and Stokes numbers can be re-expressed to consider such behaviour as:

$$Stk_{50}^*(r)Eu = 0.006 \left( \ln \frac{1}{R_f} \right)^{2.37} e^{(6.84C)} \quad (29)$$

$$Eu = 1686 (Re^*)^{-0.035} e^{(-3.39C)} \quad (30)$$

$$R_f = 32.8 \left( \frac{D_u}{D_c} \right)^{1.53} (Re^*)^{-0.34} e^{(3.70C)} \quad (31)$$

where  $Stk_{50}^*(r)$  and  $Re^*$  are the “generalized” Stokes and Reynolds numbers, meaning that they include the parameters of characterization of non-Newtonian suspensions. They may be defined as follows [20]:

$$Re^* = \frac{D^n v^{2-n} \rho}{K 8^{n-1} \left( \frac{3n+1}{4n} \right)^n} \quad (32)$$

$$Stk_{50}^*(r) = \frac{x_{50}^{n'+1}(r)(\rho_s - \rho)v^{2-n'}}{18K(3)^{n-1} \left( \frac{2n+1}{3n} \right)^n D_c} \quad (33)$$

where  $x_{50}(r)$  represents the “reduced” cut size including the previously described dead flux effect.

In terms of biological fluids and suspensions, the standard application for small hydrocyclones is for starch refining, and they have been extensively used in corn and potato starch refining giving good results for both cases [26]. Hydrocyclones have been used to separate gossypol from cottonseed protein in cottonseed oil processing. They have also been used as separators in multi-stage mixer-separator extraction systems for solu-

ble coffee. Hydrocyclones have been employed in thickening of wastewater sludge with promising results [27, 28]. Sephadex, yeasts and blood cells have been separated using hydrocyclones, too [29]. Yeast recovery in different fermentation processes has been also performed well by hydrocyclones [30–32]. Another application reported is that of recycling kieselguhr for filters in the brewing industry [33].

The dimensionless scale-up model previously described has been tested in applications of apple juice clarification [11] and wastewater treatment [28]. For the juice clarification, low values of Reynolds number were obtained due to the high viscosity of the feed, so the flow was not as turbulent as it is commonly found in a small-diameter hydrocyclone operation. The Euler number values were practically constant, which may also be attributed to the high consistency of the feed suspensions. For these reasons, relationships of  $Eu$  against  $Re$  were not properly derived. It is believed that the behaviour of the feed suspension was not adequately approached in terms of using an appropriate pump. A higher capacity slurry pump for viscous fluids could have provided better pressure drops with the consequent effect in having more defined cut sizes according to the experienced pressures and obtaining more variability in Euler and Reynolds numbers. Despite all these mentioned difficulties, from experimental data of a relationship similar to Eq. (25) was obtained [11] as follows:

$$Stk_{50}(r) = 7 \times 10^{-5} (1 - R_f) e^{(-6.47C)} \quad (34)$$

As can be seen, the constant values in Eqs. (25) and (34) show remarkable similarity. Apart from the opposite trend in the concentration correction factor, such values are practically similar. Taking into account that the conditions of derivation of both equations were totally different, i.e. inert against biological material and different hydrocyclone diameter and operating variables, the good comparison gives reasons to believe that the dimensionless approach may be extended to separation of biological materials. With regard to the wastewater case [28], practically all the feed suspensions were pseudoplastic. Therefore, a series of dimensionless relationships like those described by ORTEGA-RIVAS and SVAROVSKY [34] were attempted to be formulated. The developed correlations are presented below:

$$Eu = 3.388 \times 10^9 (Re^*)^{-1.542} e^{(-1.325C)} \quad (35)$$

$$R_f = 59.326 \left( \frac{D_u}{D_c} \right)^{4.182} (Re^*)^{-0.269} \quad (36)$$

Apart from the fact that there was no significant effect of the concentration in Eq. (35), both expressions showed adequate similarity to those suggested elsewhere [24, 34]. This may be considered an important verification of the applicability of the dimensionless scale-up model of hydrocyclone operation, due to the use of real food systems in the study.

## FILTRATION

Filtration may be defined as the unit operation in which the insoluble solid component of a solid-liquid suspension is separated from the liquid component by passing the suspension through a porous barrier, which retains the solid particles on its upstream surface, or within its structure, or both. The solid-liquid suspension is known as the feed slurry or prefilter, the liquid component that passes through the membrane is called the filtrate, and the barrier itself is referred to as the filter medium. The separated solids are known as the filter cake, once they form a detectable layer covering the upstream surface of the medium. The flow of filtrate may be caused by several means. Pressure and vacuum are two conventional ways of driving the suspension across the medium. In general terms, filtration theory applies to cases where cake builds up. Some of the more fundamental treatments of filtration theory are reviewed by WAKEMAN and TARLETON [35]. In the initial stages of filtration, the first particles of solid that encounter the filter medium become enmeshed in it, reducing its open surface area and increasing the resistance it offers to the flow of filtrate. As filtration proceeds, a layer of solids builds up on the upstream face of the medium and this layer, or cake, increases in thickness with time. Once formed, this cake in fact becomes the primary filtering medium. Filtrate passing through the filter encounters three types of resistance: the first resistance offered by channels of the filter itself, the second because of the filter medium presence, and the third due to the filter cake. The total pressure drop across the filter is equivalent to the sum of the pressure drops resulting from these three resistances. The pressure drop due to the channels of the filter is usually neglected in calculations. If  $-\Delta P$  is the total pressure drop across the filter and  $-\Delta P_c$  and  $-\Delta P_m$  the pressure drops across the cake and medium respectively, then:

$$-\Delta P = -\Delta P_c - \Delta P_m \quad (37)$$

The pressure drop across the filter cake may be

related to the filtrate flow by the following expression [8]:

$$-\Delta P_c = \frac{\alpha \mu w V}{A^2} \left( \frac{dV}{dt} \right) \quad (38)$$

where  $\alpha$  is the specific resistance of the cake,  $\mu$  is the viscosity of filtrate,  $w$  is the mass of solids deposited on the medium per unit volume of filtrate,  $V$  is the volume of filtrate, and  $A$  is the filter area normal to the direction of filtrate flow.

If a cake is composed of rigid non-deformable solid particles,  $\alpha$  is independent of  $-\Delta P_c$  and does not vary throughout the depth of the cake. This type of cake is known as incompressible cake. However, if the cake contains non-rigid, deformable solid particles or agglomerates of particles, the resistance to flow will depend on the pressure drop and will vary throughout the depth of the cake. In this case, the cake is called compressible and an average value of the specific resistance for the entire cake must be used in Eq. (38). This average specific resistance must be measured experimentally for any particular slurry.

By analogy with Eq. (38), the filter medium resistance may be defined by the following relation:

$$-\Delta P_m = \frac{R_m \mu}{A} \left( \frac{dV}{dt} \right) \quad (39)$$

where  $-\Delta P_m$  is the pressure drop across the medium and  $R_m$  is the filter medium resistance.

It is reasonable to assume that  $R_m$  is constant during any filtration cycle and that it includes the resistance to filtrate flow offered by the filter channels. In this case, Eqs. (37), (38) and (39) can be combined to give:

$$\frac{dV}{dt} = \frac{A(-\Delta P)}{\mu \left( \frac{\alpha w V}{A} + R_m \right)} \quad (40)$$

Eq. (40) is a general expression for the filtrate flow rate.

When the pressure drop is maintained constant, Eq. (40) may be integrated thus:

$$\int_0^t dt = \frac{\mu}{A(-\Delta P)} \left( \frac{\alpha w}{A} \int_0^V V dV + R_m \int_0^V dV \right) \quad (41)$$

or, substituting limits and transposing for time  $t$ :

$$t = \frac{\mu}{(-\Delta P)} \left[ \frac{\alpha w}{2} \left( \frac{V}{A} \right)^2 + R_m \left( \frac{V}{A} \right) \right] \quad (42)$$

Eq. (42) is a general expression for the filtration time during constant pressure filtration. In

order to use it, values of  $\alpha$  and  $R_m$  must be determined experimentally. This can be done by re-writing it in the following form:

$$\frac{dt}{dV} = K V + B \quad (43)$$

where:

$$K = \left( \frac{\alpha w \mu}{A^2 (-\Delta P)} \right) \quad (44)$$

and:

$$B = \left( \frac{R_m \mu}{A (-\Delta P)} \right) \quad (45)$$

As can be gathered, Eq. (43) represents a straight line if  $dt/dV$  is plotted against  $V$ . Therefore, if a constant pressure filtration is carried out and values of  $V$  for different values of  $t$  are recorded, a graph of  $dt/dV$  versus  $V$  can be constructed. The slope of this line is  $K$  and the intercept on the ordinate when  $V = 0$  is  $B$ . Thus, by using such a graph, values of  $\alpha$  and  $R_m$  can be directly determined from Eqs. (44) and (45).

If filtration is carried out at a constant rate, then:

$$\frac{dV}{dt} = \text{constant} = \frac{V}{t} \quad (46)$$

Eq. (40) may be re-written as follows:

$$-\Delta P = \left( \frac{\mu \alpha w V}{A^2 t} \right) V + \left( \frac{\mu V R_m}{A t} \right) \quad (47)$$

or:

$$-\Delta P = K' V + B' \quad (48)$$

Once again it can be seen that Eq. (48) represents a straight line if  $-\Delta P$  is plotted against  $V$ .

The slope of the line is  $K'$  and the intercept to the  $-\Delta P$  axis when  $V = 0$  is  $B'$ . Thus, for incompressible cakes  $\alpha$  and  $R_m$  can again be determined by experimental means. Eq. (47) can then be used for cycle calculations [36].

Although filtration theory has been well established and tested, it was developed for areas different from food application. For filtration in food processing, adapted models had to be suggested. Filtration of beverages and wine have been attempted to be described by models such as that represented by Eq. (40) [37], but the success was only limited. DE LA GARZA and BOULTON [38] modified such model for wine filtrations, proposing the following exponential equation:

$$\frac{dV}{dt} = \frac{(-\Delta P)A}{\mu} \cdot \frac{1}{R_m e^{(kV/A)}} \quad (49)$$

where  $k$  is a positive constant.

Prior to the wide commercialization of membrane separations, conventional filtration was the final clarification step in many food applications, particularly in the production of fruit juices and fermented products, such as beer and wines. Similarly to the case of centrifugation, utilization of filtration has focused on quality aspects, not on microbial safety. Polyphenol oxidase (PPO) particulate fractions have been identified as responsible for enzymatic browning in fruit juices such as apple, pear and grape juices. Those fractions may be removed by conventional filtration using Bentonite or other filter aids. SAPERS [10] reported on investigation of effects of filtering fruit juices under suction with different filter aids. Granny Smith and Golden Delicious apple juices, filtered with addition of a diatomaceous earth as filter aid, prevented enzymatic browning during subsequent storage. BAYINDIRLI et al. [37] fitted satisfactorily the De La Garza and Boulton's exponential filtration model, originally developed for wine filtration, to apple juice filtration. The parameters they found coincided with those of the De La Garza and Boulton's model, and they used them to interpret the effects of various operation variables on filtration rates. They also tried to exploit the SPERRY's [39] model described by Eq. (40), but the results were not satisfactory.

SAHIN and BAYINDIRLI [40] presented another investigation using De La Garza and Boulton's exponential model for cake filtration of sour cherry juice, successfully fitting it to such an application. They found an inverse relation between the filtration rate and the filtration resistance. They also looked at the effects of increasing the amount of precoating, which decreased the filtration rate. On the other hand, an increase in filter aid dose increased the filtration rate.

MAXIMEA and LAMELOISE [41] presented results of studies on filterability of viscous complex food salting solutions. They sought for a suitable coupling for the regeneration of complex concentrated ternary brine after fish fillet salting using filtration. Three selected waste brines from different clean solutions and salting process conditions were used. Processing plant considerations, such as economics, environment and technical availabilities were taken into account. Several chemical and physical pre-treatments were first conducted to increase the size of suspended solids and to reduce the proteinaceous matters in the suspension.

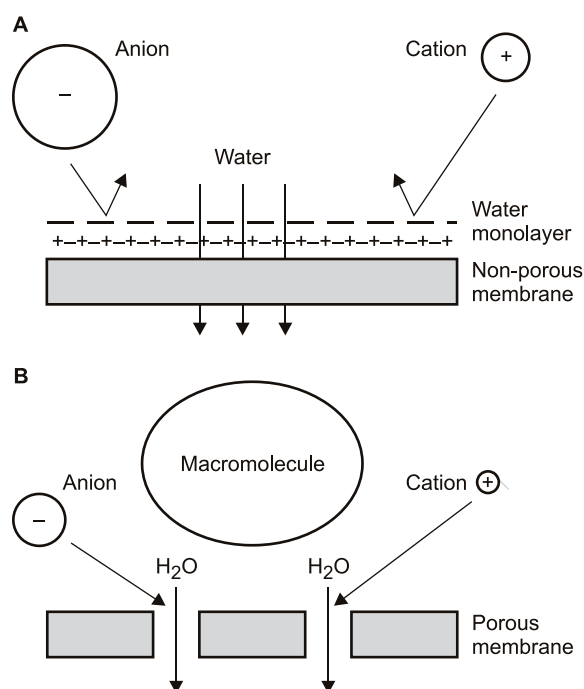
Treated suspensions and their separated phases after settling were subjected to various filtration experiments. Laboratory- and industrial-scale filtration experiments using a rotary vacuum precoat filter were conducted to find the optimal filtration conditions. The best coupling appeared to be pH pretreatment, followed by a settling time adjusted to the salting process conditions.

Filtration has also been used as an aid for pasteurization or sterilization. REDDY et al. [42] produced sterilized green coconut water by using constant pressure filtration on a two-stage laboratory set-up. The first step employed ordinary filter paper for removal of suspended particles, while the second one made use of a microfiltration cellulose nitrate membrane for the removal of microorganisms. Three pressure levels were tested and resistances for filtration process and compressibility factor were determined. Both filter medium and cake resistances increased with an increase in applied pressure differentials. Although filter medium resistance values for corresponding pressure differentials were high for the membrane filter, the values of cake resistance were comparable for both systems. The deposited cakes were compressible. Investigations on inactivation of vegetative cell suspensions (*Escherichia coli* and *Salmonella typhimurium*) and spore suspensions (*Bacillus subtilis* and *Bacillus licheniformis*) by hydrostatic pressure treatment have been reported [43]. Inactivation ratios between the filtered and unfiltered vegetative cell suspensions were not different. Filtered spore suspensions were inactivated easier than the unfiltered ones. Filtered *B. subtilis* spores were sterilized in 90 min while the unfiltered spores were inactivated in 180 min. The authors concluded that the filtration of spore suspensions was effective to increase the inactivation ratio by the hydrostatic pressure treatment, but did not contribute to increase the inactivation ratio of vegetative cell suspensions.

## MEMBRANE SEPARATIONS

Membrane separations are techniques used industrially for removal of solutes and emulsified substances from solutions by application of pressure onto a very thin layer of a substance with microscopic pores, known as a membrane. Membrane separation processes include reverse osmosis (RO), ultrafiltration (UF), microfiltration (MF), dialysis, electrodialysis, gas separation, and pervaporation. RO, UF, MF and electrodialysis have been widely used commercially [44]. UF has found many applications in food





**Fig. 8.** Reject mechanisms for reverse osmosis (A) and ultrafiltration (B).

processing and has been successfully employed in a number of liquid “cold sterilization” and clarification applications. For the case of UF, membrane pore size ratings are generally in the range of 0.001–0.020  $\mu\text{m}$  [45].

Mechanisms of separation of UF and RO are illustrated in Fig. 8. In RO, a rejection is based on electrostatic repulsion due to formation of a pure layer of water over the membrane, and the virtual charges on this layer reject charges of ionic free species of salt solutions. Simultaneously, by a complex mechanism of sorption, diffusion and desorption, pure water passes through the membrane performing the separation process. UF membranes are porous in nature, with a rigid and highly voided structure, and function in a manner analogous to a screen or sieve (Fig. 8B). The pore network is randomly distributed, with pores passing directly through the membrane. The separating ability is based primarily on particle size, wherein particles and molecules larger than the largest pore are completely retained, whereas species smaller than the smallest pore are totally permeated. Therefore UF is an extension of conventional filtration, with the separating ability extended to the molecular level.

The molecular weight rating of an UF membrane is expressed in terms of a rejection coefficient against a species of specific molecular weight

[45]. Ideally, the membranes will have a sharply defined molecular weight cut-off (MWCO), as illustrated in Fig. 9. Such an ideal membrane will retain all species greater than the MWCO but will allow all smaller ones to pass. Membranes are available in a number of increments in MWCO, ranging from 1000 up to 100000 Daltons (Da). The importance to food applications lies in the fact that these membrane characteristics give specificity in terms of permeating soluble saccharides and flavour components, and retaining suspended solids, microorganisms, spores and other particulates responsible for spoilage.

The flow rate  $Q$  through a membrane separation process may be represented by [46]:

$$Q = kA (\Delta P - \Delta \pi) \quad (50)$$

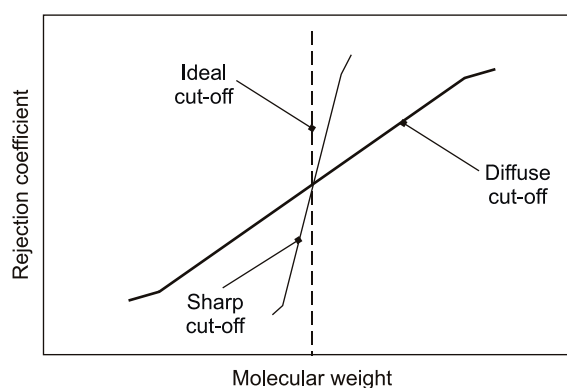
where  $k$  is a membrane permeability coefficient,  $A$  is the membrane superficial area,  $\Delta P$  is the pressure drop, and  $\Delta \pi$  is the difference in osmotic pressure between the feed and the permeate.

Eq. (50) is more suitable for RO applications, due to the osmotic pressure exerted by solutes in such applications. For UF applications,  $\Delta \pi$  is negligible in relation to  $\Delta P$  and the membrane thickness causes an effect in the process, so it should be included as a variable. Under these considerations, Eq. (50) can be re-expressed as:

$$Q = \left( \frac{kA}{\delta} \right) \Delta P \quad (51)$$

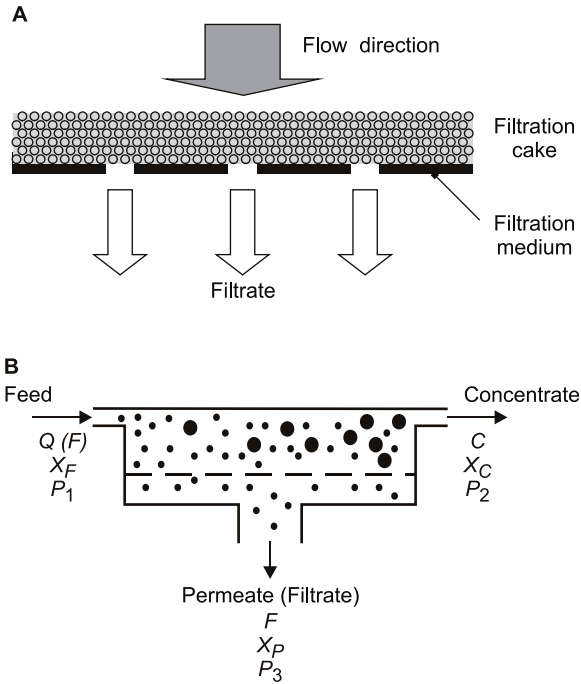
where  $\delta$  is the membrane thickness.

The pressure drop  $\Delta P$  in Eq. (51) is, really, an average pressure or pressure gradient due to the cross-flow arrangement of membrane separation processes. A feed stream containing solids of different size is pumped across the membrane surface at a velocity determined by the feed side



**Fig. 9.** Representation of molecular weight cut-off.





**Fig. 10.** Mode of operation of ultrafiltration membrane.

A – Dead-end filtration, B – Cross-flow filtration, with mass balance.

pressure gradient. This gradient, known as hydrodynamic pressure gradient, causes the continuous movement of fluid across the membrane that is referred to as cross-flow. As the feed stream flows across the membrane surface, smaller particles may be able to pass through the membrane and exit in the permeate stream at a pressure, which is usually atmospheric. The rate of permeate flow is generally reported as flux, i.e. flow rate per unit area of membrane. The driving force for permeate flow is also a pressure gradient, but is not the hydrodynamic pressure gradient defined above. Instead, it is the pressure gradient that exists through the membrane, from feed size to permeate size, at each point along the membrane surface. This pressure gradient is known as the trans-membrane pressure gradient or simply trans-membrane pressure ( $TMP$ ). Clearly,  $TMP$  varies along the membrane surface being maximal at the inlet and minimal at the outlet [47]. An average  $TMP$  can be defined as (Fig. 10):

$$TMP = \frac{P_1 + P_2}{2} - P_3 \quad (52)$$

The permeate pressure  $P_3$  is negligible compared to the pressure gradient between feed and concentrate. Thus:

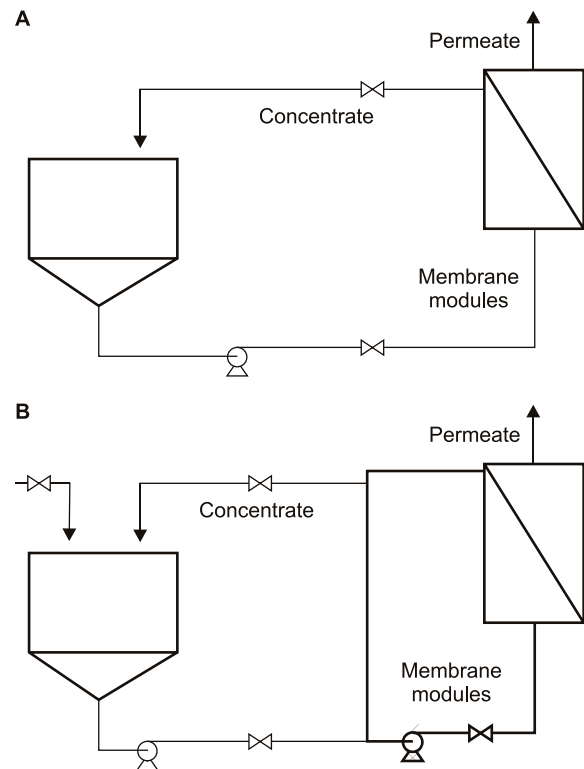
$$TMP = \frac{P_1 + P_2}{2} \quad (53)$$

A way of expressing efficiency of a membrane separation process is by determining a recovery percentage as the relation of the permeate flow rate  $F$  to the feed flow rate  $Q$ , i.e.:

$$\text{Recovery [\%]} = \frac{F}{Q} \cdot 100 \quad (54)$$

Recovery percentage depends on the feed concentration, being higher at lower concentrations of feed solids. A fraction of liquid, approximately equal to that of solids, should remain in the concentrate to make it flow. Therefore, the more diluted the feed, the higher the percentage of recovery.

UF may be considered to be an extension of conventional filtration, but the operating mode of the equipment is different. In UF (Fig. 10), the fluid moves continuously across the membrane surface (cross-flow filtration) and is used as means of sweeping the membrane surface to control the build-up of foulants. UF may be integrated in processing lines in several ways. The



**Fig. 11.** Ultrafiltration systems.

A – Standard batch configuration, B – Topped-off batch system with recirculation loop.

simplest technique is the batch configuration illustrated in Fig. 11A. In this mode, an initial volume of liquid is circulating through the UF system, and the permeate is continuously removed until the final volume is achieved. Re-circulating topped-off batch is a variation shown in Fig. 11B.

Dairy processing has been a traditional application of membrane separations. Concentration of whole milk by membranes has been used for different purposes. For example, whole milk was concentrated using a 50 000 Da pore membranes, and the effects on retention of total solids, proteins and fat content, were studied [48]. The membrane satisfactorily retained the main nutrients in the concentrate. In whey processing, which represents one of the first fields of application of membrane processes in the dairy industry [49, 50], MF has been used to reduce the total number of lactic acid bacteria [51] and other microorganisms [52, 53] in whey. It has been reported that the process also resulted in de-fatting of the whey and was considered as a gentle sterilization method [54, 55]. The performance of UF and nanofiltration (NF) membranes was investigated at concentration of whey proteins and lactose [56]. Permeate flux, membrane retention and yield were determined at varying pressure, recycle flow rate and temperature. Effects on whey proteins and lactose concentration were determined. The permeate flux, protein and lactose contents in permeate and concentrate fractions were measured during the experimental runs. The membranes tested were found suitable for concentration of milk, as well as the whey proteins and lactose with high flux and retention. A vibratory shear-enhanced filtration system was used for the separation of casein micelles from whey proteins of skim milk reconstituted from low heat-treated milk powder with similar protein content as fresh milk [57]. The performances of MF with polytetrafluoroethylene (PTFE) membrane and UF with polyethersulfone membrane were compared. The critical flux for stable operation was investigated in MF by increasing the permeate flux in steps, while monitoring the trans-membrane pressure. The UF membrane minimized casein loss in the permeate with a little reduction in permeate flux. For casein micelles, at separation from whey proteins with shear-enhanced filtration, the use of a UF membrane with 150 kDa cut-off allowed to minimize micelle loss while producing acceptable whey proteins.

The dairy industry produces high amounts of effluents and wastewaters, and membrane separations have also found applications in downstream processing. For example, permeate flux and

chemical oxygen demand reductions were investigated in dairy processing waters using different NF and RO membranes [52]. In NF, the highest permeate flux was obtained with a particular membrane type, which yielded also the highest permeate chemical oxygen demand. In concentration tests, the permeate flux decreased with increasing volume reduction ratio while the chemical oxygen demand of the permeate increased. The performance of dairy process waters treatment with membranes for recovery of milk constituents and water were investigated in terms of chemical oxygen demand and ion rejection [58]. Some dead-end filtration experiments allowed for comparison of NF and RO membranes. A single membrane operation was insufficient for producing water of composition complying with the requirements for drinking water. Chemical oxygen demand and milk ions concentration in the permeate remained too high even with RO membranes.

UF can be used as a unique operation for clarification and pasteurization of fruit juices, such as apple juice, due to its operating principle [59]. Some investigations have been reported regarding quality of apple juice treated by membrane filtration. HEATHERBELL et al. [60] clarified apple juice by UF and obtained a stable clear product. RAO et al. [61] studied retention of odour-active volatiles using different UF membrane materials. PADILLA and McLELLAN [62] investigated the effect of MWCO of UF membranes on quality and stability of apple juice. Comparison of microfiltration and UF has also been done [63], and it has been found that the microfiltered juice contained more soluble solids and it was more turbid compared to the ultrafiltered juice. The use of mineral membranes for apple juice clarification has also been reported [64]. In general terms, the use of UF to clarify apple juice resulted in fresh appearance because the product was cold-sterilized, avoiding undesirable reactions triggered by the conventional thermal process. As with common pasteurization, it has been stated that the advantage of thermal processing is the inactivation of enzymes that cause browning of juices [10]. However, since it has also been suggested [65] that enzyme activity is associated with particulate fractions, UF might also be used to separate such particulates from raw apple juice in order to prevent, or greatly reduce, enzymatic browning.

Juices produced by membrane filtration and traditional methods have mostly been shown to have similar properties. HEATHERBELL et al. [60] compared apple juice clarified by UF and gelatin fining. The juice fined by gelatin was pasteurized before fining, which reduced oxidative reactions

leading to colour development. Bottled non-pasteurised UF juice was informally panel-tested and was reported to have superior flavour compared to canned UF or canned gelatin-fined juice. However, the bottled juice did develop a sediment after storage. Slight differences have also been noted between membrane-clarified and traditionally clarified juices. RAO et al. [61] reported that the retention of odour-active volatiles in UF apple juice was intermediate compared to juices prepared by traditional plate and frame filtration or vacuum drum filtration. Plate and frame filtration gave the highest retention. RWABAHIZI and WROLSTAD [66] found that strawberry juice clarified by filtration through a 10kDa hollow fibre membrane lost on average 55% anthocyanins compared to 17% loss at conventional filtration. DRAKE and NELSON [67] compared commercial apple juice made by UF at 50kDa MWCO at 75°C and pasteurized at 85°C, with commercial juices produced using a conventional plate and frame filtration system. The UF juice had lower turbidity, 5% higher soluble solids and lesser colour than the other juice. It was also sensorically rated as lighter in colour with more of the initial watery mouth-feel.

Some studies have demonstrated that membrane material may have some influence on UF and MF processing. BRADDOCK [68] used UF and RO to recover limonene from citrus processing waste streams. Membrane flux rates declined after the contact with limonene (around 0.11%). Polysulfone membranes had the most severe decline followed by cellulose acetate and PTFE. RAO et al. [61] studied the retention of eight odour-active volatiles in apple juice filtered through a 50 kDa polysulfone and a 30-kDa polyamide hollow fibre membrane. The permeate from the polyamide membrane contained more volatiles than that from the polysulfone membrane. BEN AMAR et al. [58] found that a 0.2µm ceramic membrane and a 0.2µm carbon membrane gave similar fluxes for apple juice under similar conditions. CAPANNELLI et al. [69] tested polysulfone, polyvinylidene fluoride and ceramic membranes of various MWCO with orange and lemon juices. At given working conditions, the flux was largely independent of membrane material and MWCO. This was attributed to fibrous deposits that developed at the membrane surfaces and acted as a dynamic semi-permeable barrier. RIEDL et al. [70] examined how membrane structure influenced the aggregation of apple juice colloidal particles on porous MF membrane surfaces. Using atomic force microscopy, polyethersulfone and polyvinylidene fluoride membranes were found to have rough surface structures that pro-

duced looser surface fouling layers than the dense fouling layers observed on smooth-surfaced membranes such as polyamide and polysulfone.

HEATHERBELL et al. [60] filtered apple juice with a 50kDa MWCO polysulfone membrane to a twofold concentration. The permeate had low microbial counts, while the concentrate satisfactorily retained microorganisms. No evidence of spoilage was detected in aseptically bottled product after storage at room temperature for 6 months. GROHMANN and FEUERPEIL [71] mentioned that ceramic membranes that can be thermally sterilized provide maximum operating safety in the production of a cold-sterilized juice using tangential flow systems. The authors were able to produce a cold-sterilized apple juice with a 0.2µm ceramic membrane on a large scale. With polymeric membranes, minimal operating pressures should also be used to prevent damage and thus occurrence of contamination. Cross-flow filtration can be combined with a final dead-end filtration to commercially produce cold-sterilized juice products and ensure microbiological stability. REID et al. [72] described a set-up for aseptic filtration and bottling of beer into polyethylene terephthalate containers. The bottle rinsing, filling and capping operations were maintained in a filtered air environment to minimize microbial contamination. TRONC et al. [73] used bipolar membranes to lower the pH of cloudy apple juice. By circulating apple juice on the cationic side of a bipolar membrane, pH was reduced from 3.5 to 2.0, which completely inhibited PPO activity compared to the control. Following this temporary acidification, pH was returned to its initial value by re-circulating the juice on the anionic part of the bipolar membrane. Although the pH re-adjustment partially reactivated PPO, the colour of the cloudy apple juice remained stable during storage. In acidic media, the free carboxyl groups of amino acids are protonated and the negative charges are neutralized. Inhibition of PPO likely resulted from the change in the tertiary structure of the proteins caused by electrostatic repulsion between acids and positively charged amino groups [74, 75].

ZARATE-RODRIGUEZ et al. [76] presented a study on the effects of the pore size of UF membranes on the quality of the treated apple juice. Fresh apple juice was processed using an UF unit with polysulphone membranes of 10kDa and 50kDa pore sizes. Transmembrane pressures of 103, 120.5, 138 and 155 kPa were used. Recovery percentages of 0, 25, 50 and 75 were tested for the smaller pore membrane, and 0, 10, 20, 30, 40, 50 and 60 for the larger pore membrane. The responses to these factors were evaluated for the

following quality attributes: pH, acid content, soluble solids and colour. In general terms, pH, acid content and soluble solids did not change but presented less variability for the smaller pore membrane treatment.

## CONCLUSIONS

Solid-liquid separations have been extensively used in the food industry, mainly as a final stage for purification of liquids or for recovering of solids. These separation technologies had been considered as mechanical operations and had been focused only on quality improvement. The development and scaling-up of membrane separations to allow their wide commercialization led to the perception of solid-liquid separations as transformation technologies. Non-thermal pasteurization of various liquid food systems is now industrially used at various alternative arrangements of solid-liquid separations. The microbial safety, nutritive quality and sensory value of different liquid foods may be guaranteed by means of processing operations based, totally or partially, on solid-liquid separation technologies.

## Notation

Dimensions given in terms of mass ( $M$ ), length ( $L$ ), time ( $T$ ), and temperature ( $\theta$ ).

$A$	Area ( $L^2$ )
$B$	Intercept with y-axis in plot of constant pressure filtration run ( $T/L^3$ )
$B'$	Intercept with y-axis in plot of constant rate filtration run ( $M/LT^2$ )
$b$	Height of bowl of tubular centrifuge ( $L$ )
$C$	Volume fraction of solids in suspension
$C_D$	Drag coefficient
$D_c$	Hydrocyclone diameter ( $L$ )
$D_i$	Inlet diameter of hydrocyclone ( $L$ )
$D_o$	Overflow pipe diameter of hydrocyclone ( $L$ )
$D_u$	Underflow diameter of hydrocyclone ( $L$ )
$E_p$	Partial efficiency
$E_t$	Total efficiency
$Eu$	Euler number
$f(x)$	Frequency or occurrence related to size
$F$	Permeate flow rate ( $L^3/T$ )
$F(x)$	Cumulative frequency
$F_D$	drag force ( $ML/T^2$ )
$g$	Acceleration due to gravity ( $L/T^2$ )
$G(x)$	Grade efficiency
$G'(x)$	Reduced grade efficiency
$k$	Constant
$k_p$	Constant for a family of geometrically similar hydrocyclones
$k_1, k_2$	Constants

$K$	Constant, correlation constant of the power-law, slope of line in plot of constant pressure filtration run ( $T/L^6$ )
$K'$	Slope of line in plot of constant rate filtration run ( $M/L^4T^2$ )
$L$	Length ( $L$ )
$M$	Mass flows rate of solids in suspension ( $M/T$ )
$M_c$	Mass flows rate of separated solids ( $M/T$ )
$M_f$	Mass flows rate of unseparated solids ( $M/T$ )
$n$	Flow behaviour index
$n_p$	Constant for a family of geometrically similar hydrocyclones
$P$	Pressure ( $M/LT^2$ )
$Q$	Volumetric flow rate ( $L^3/T$ ), feed flow rate ( $L^3/T$ )
$R$	Radius ( $L$ )
$Re$	Reynolds number
$Re_p$	Particle Reynolds number
$Re^*$	Reynolds number for power-law fluids
$R_f$	Underflow-to-throughput ratio
$R_m$	Resistance of filter medium ( $1/L$ )
$R_x$	Outer radii of stack of discs in disc-bowl centrifuge ( $L$ )
$R_y$	Inner radii of stack of discs in disc-bowl centrifuge ( $L$ )
$S$	Number of discs in stack in disc-bowl centrifuge
$Stk_{50}$	Stokes number
$Stk^*_{50}$	Stokes number for power-law fluids
$Stk^*_{50}(r)$	Stokes number for power-law fluids including the reduced cut size
$t$	Time ( $T$ )
$TMP$	Trans-membrane pressure ( $M/LT^2$ )
$u$	Linear velocity in vertical direction ( $L/T$ )
$u_g$	Terminal settling velocity of particles ( $L/T$ )
$u_t$	Terminal settling velocity of particles ( $L/T$ )
$v$	Linear velocity in horizontal direction ( $L/T$ )
$v_g$	Terminal settling velocity under gravity ( $L/T$ )
$v_r$	Radial settling velocity ( $L/T$ )
$V$	Volume ( $L^3$ )
$w$	Mass of solids deposited on the medium per unit volume of filtrate ( $M/L^3$ )
$x$	Particle size
$x_c$	Cut point ( $L$ )
$x_{50}$	Cut size ( $L$ )
$x_{50}(r)$	Reduced cut size ( $L$ )

## Greek letters

$\alpha$	Specific cake resistance ( $L/M$ )
$\gamma$	Shear rate ( $1/T$ )
$\delta$	Membrane thickness ( $L$ )
$\Delta P$	Pressure drop ( $M/LT^2$ ); pressure drop across fixed bed ( $M/LT^2$ )
$-\Delta P$	Total pressure drop across filter ( $M/LT^2$ )
$-\Delta P_c$	Pressure drops across the cake ( $M/LT^2$ )
$-\Delta P_m$	Pressure drops across the medium ( $M/LT^2$ )
$\mu$	Liquid absolute viscosity ( $M/LT$ ), suspension viscosity ( $M/LT$ )
$\pi$	Osmotic pressure ( $M/LT^2$ )
$\rho$	Liquid density ( $M/L^3$ )
$\rho_s$	Solids density ( $M/L^3$ )



$\Sigma$	Characteristic geometrical features of a centrifuge equivalent to area of a gravity settling tank with similar settling characteristics of a centrifuge
$\tau$	Shear stress ( $M/LT^2$ )
$w$	Angular velocity ( $1/T$ )
$W$	Conical half angle of discs in a disc-bowl centrifuge

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