

## 15 Mixing, Emulsification and Size Reduction

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### 15.1 Mixing (Agitation, Blending)

#### 15.1.1 Introduction

Mixing is a unit operation widely used in food processing. Many definitions of this term have been proposed. One of the simplest is: “an operation in which a uniform combination of two or more components is formed”. In addition to blending components together, mixing operations may bring about other desirable changes in the materials being mixed, such as mechanical working (as in dough mixing), promotion of heat transfer (as in freezing ice cream) facilitating chemical or biological reactions (as in fermentation). The components in a mixing operation may be liquids, pastes, dry solids or gases. The degree of uniformity attainable in a mixing operation varies, depending on the nature of the components. In the case of low viscosity miscible liquids or highly soluble solids in liquids a high degree of uniformity is attainable. Less intimate mixing is likely to occur in the case of viscous liquids, pastes and dry solids. Combining immiscible materials together usually requires specialised equipment, which is covered under emulsification in Section 15.2. Efficient utilisation of energy is another criterion of mixing. This is more easily attainable in the case of low viscosity liquids as compared with pastes and dry solids.

#### 15.1.2 Mixing of Low and Moderate Viscosity Liquids

The impeller mixer is the most commonly used type of mixer for low viscosity liquids (viscosity less than 100 poise;  $10 \text{ N s m}^{-2}$ ). Such a mixer consists of one or more impellers, fixed to a rotating shaft and immersed in the liquid. As the impellers rotate, they create currents within the liquid, which travel throughout the mixing vessel. If turbulent conditions are created within the moving

streams of liquid, mixing will occur. Turbulence is usually most vigorous near the impeller and the liquid should pass through this region as often as possible. The fluid velocity in the moving streams has three components: (a) a radial component acting in a direction at right angles to the shaft, (b) a longitudinal component acting parallel to the shaft and (c) a rotational component acting in a direction tangential to the circle of rotation of the shaft. The radial and longitudinal components usually promote mixing but the rotational component may not.

If an impeller agitator is mounted on a vertical shaft located centrally in a mixing vessel, the liquid will flow in a circular path around the shaft. If laminar conditions prevail, then layers of liquid may form, the contents of the vessel rotate and mixing will be inefficient. Under these conditions a vortex may form at the surface of the liquid. As the speed of rotation of the impeller increases this vortex deepens. When the vortex gets close to the impeller, the power imparted to the liquid drops and air is sucked into the liquid. This will greatly impair the mixing capability of the mixer. Rotational flow may cause any suspended particles in the liquid to separate out under the influence of centrifugal force. Rotational flow, and hence vortexing, may be reduced by locating the mixer offcentre in the mixing vessel and/or by the use of baffles. Baffles usually consist of vertical strips fixed at right angles to the inner wall of the mixing vessel. These break up the rotational flow pattern and promote better mixing (Fig. 15.1). Usually four baffles are used, with widths corresponding to 1/18th (5.55%) to 1/12th (8.33%) of the vessel diameter.

Three main types of impeller mixers are used for liquid mixing.

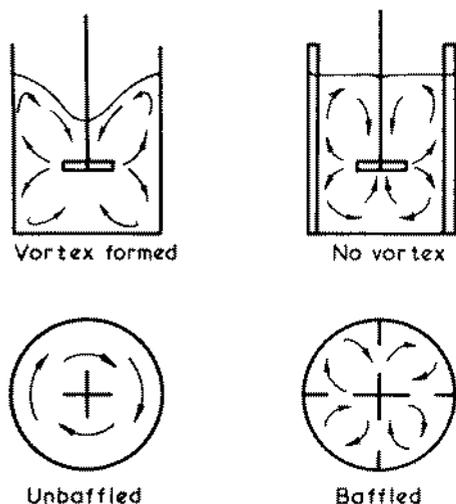


Fig. 15.1 Flow patterns in baffled and unbaffled vessels with paddle or turbine agitators; from [6] with permission of the authors.

### 15.1.2.1 Paddle Mixer

This type of mixer consists of a flat blade attached to a rotating shaft, which is usually located centrally in the mixing vessel (Fig. 15.2). The speed of rotation is relatively low, in the range 20–150 rpm. The blade promotes rotational and radial flow but very little vertical flow. It is usually necessary to fit baffles to the mixing vessel. Two or four blades may be fitted to the shaft.

Other forms of paddle mixer include: (a) the gate agitator (Fig. 15.2b), which is used for more viscous liquids, (b) the anchor agitator (Fig. 15.2c), which rotates close to the wall of the vessel and helps to promote heat transfer and prevent fouling in jacketed vessels and (c) counter-rotating agitators (Fig. 15.2d), which develop relatively high shear rates near the impeller. Simple paddle agitators are used mainly to mix miscible liquids and to dissolve soluble solids in liquids.

### 15.1.2.2 Turbine Mixer

A turbine mixer has four or more blades attached to the same shaft, which is usually located centrally in the mixing vessel. The blades are smaller than paddles and rotate at higher speeds, in the range 30–500 rpm. Simple vertical blades (Fig. 15.3a) promote rotational and radial flow. Some vertical flow develops when the radial currents are deflected from the vessel walls (Fig. 15.1). Swirling and vortexing are minimised with the use of baffles. Liquid circulation is generally more vigorous than that produced by paddles and shear and turbulence is high near the impeller itself.

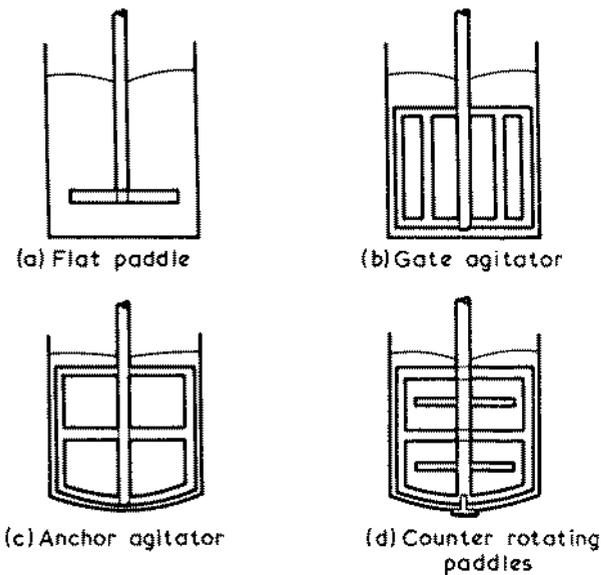


Fig. 15.2 Some typical paddle impellers; from [6] with permission of the authors.

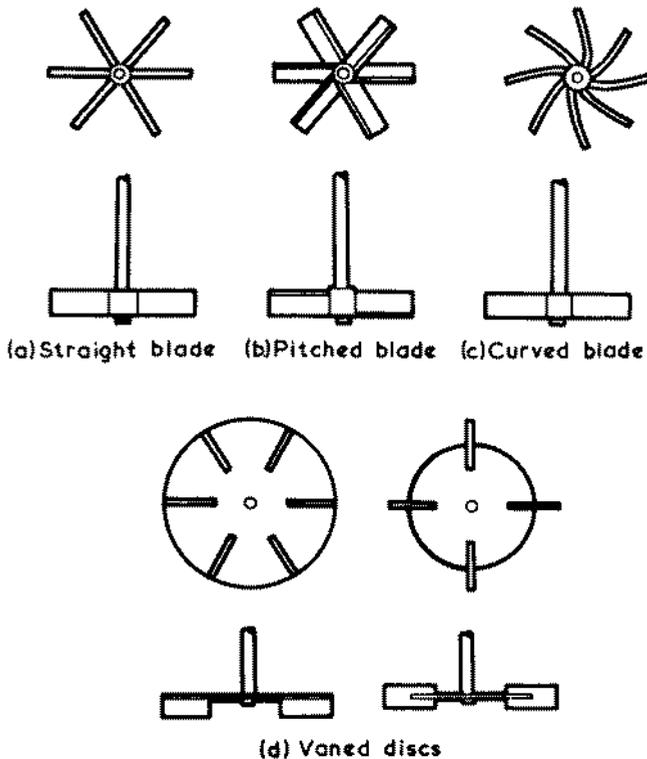


Fig. 15.3 Some typical turbine impellers; from [16] with permission of the authors.

Pitched blades (Fig. 15.3b) increase vertical flow. Curved blades (Fig. 15.3c) are used when less shear is desirable, e.g. when mixing friable solids. Vaned or shrouded discs (Fig. 15.3d) control the suction and discharge pattern of the impeller and are often used when mixing gases into liquids. Turbine mixers are used for low and moderate viscosity liquids, up to 600 poise, for preparing solutions and incorporating gases into liquids.

### 15.1.2.3 Propeller Mixer

This type of mixer consists of a relatively small impeller, similar in design to a marine propeller, which rotates at high speed, up to several thousand rpm. It develops strong longitudinal and rotational flow patterns. If mounted on a vertical shaft and located centrally in the mixing vessel, baffling is essential (Fig. 15.4a,b). Alternatively, the shaft may be located off centre in the vessel and/or at an angle to the vertical (Fig. 15.4c). When mixing low viscosity liquids, up to 20 poise, the currents developed by propeller agitators can travel throughout large vessels. In such cases the shaft may enter through the side wall of the tank (Fig. 15.4d). Special propeller designs are available which pro-

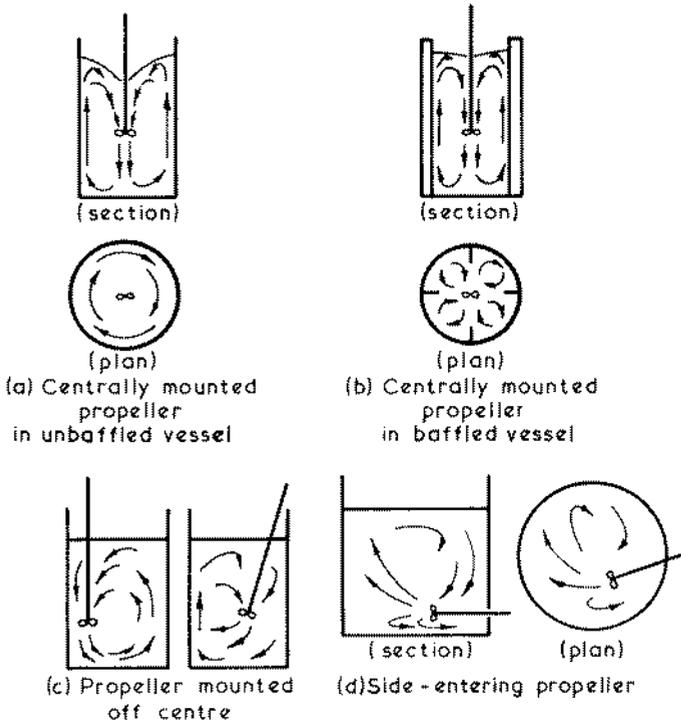


Fig. 15.4 Flow patterns in propeller agitated systems; from [16] with permission of the authors.

mote shear, for emulsion premixing. Others have serrated edges for cutting through fibrous solids.

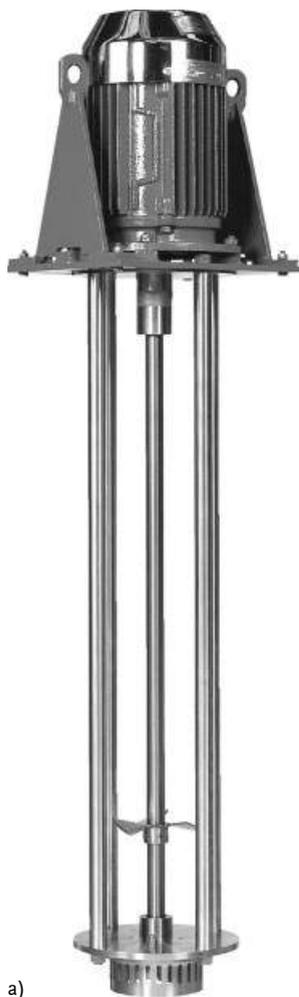
Many other types of impeller mixers are used for low viscosity liquids, including discs and cones attached to shafts. These promote gentle mixing. More specialised mixing systems are available for emulsion premixing, dispersion of solids and similar duties. One such system is shown in (Fig. 15.5).

Other methods of mixing low viscosity liquids include: (a) pumping them through pipes containing bends, baffles and/or orifice plates, (b) injecting one liquid into a tank containing the other components and (c) recirculating liquids through a holding tank using a centrifugal pump [1–8].

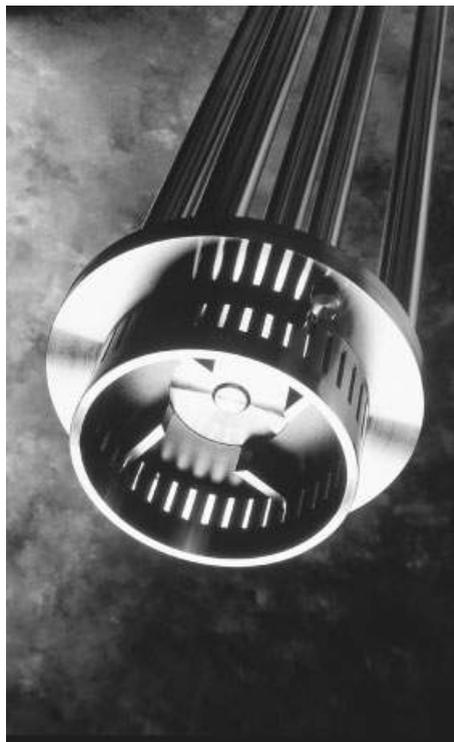
### 15.1.3

#### Mixing of High Viscosity Liquids, Pastes and Plastic Solids

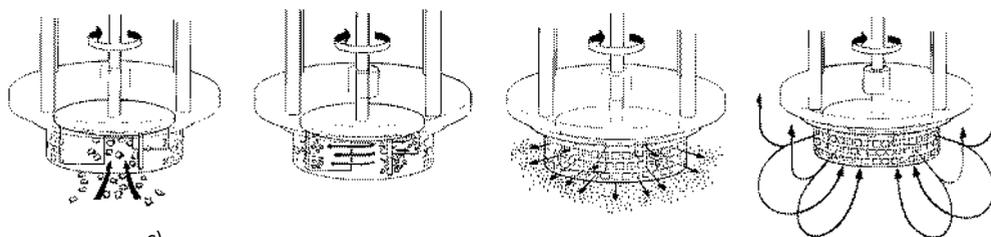
When mixing highly viscous and pastelike materials, it is not possible to create currents which will travel to all parts of the mixing vessel, as happens when mixing low viscosity liquids. Consequently, there must be direct contact between the mixing elements and the material being mixed. The mixing elements must



a)



b)



c)

**Fig. 15.5** A high shear mixer: (a) the complete mixer, (b) the mixing head, (c) high shear mixer in operation; by courtesy of Silverson Machines Ltd.

travel to all parts of the mixing vessel or the material must be brought to the mixing elements. Mixers for such viscous materials generally need to be more robust, have a smaller working capacity and have a higher power consumption than those used for liquid mixing. The speed of rotation of the mixing elements is relatively low and the mixing times long compared to those involved in mixing liquids.

#### 15.1.3.1 Paddle Mixers

Some designs of paddle mixer, of heavy construction, may be used for mixing viscous materials. These include gate, anchor and counter-rotating paddles, as shown in Fig. 15.2a, b and c, respectively.

#### 15.1.3.2 Pan (Bowl, Can) Mixers

In one type of pan mixer the bowl rests on a turntable, which rotates. One or more mixing elements are held in a rotating head and located near the bowl wall. They rotate in the opposite direction to the pan. As the pan rotates it brings the material into contact with the mixing elements. In another design of pan mixer, the bowl is stationary. The mixing elements rotate and move in a planetary pattern, thus repeatedly visiting all parts of the bowl. The mixing elements are shaped to pass with a small clearance between the wall and bottom of the mixing vessel (Fig. 15.6). Various designs of mixing elements are used, including gates, forks, hooks and whisks, for different applications.

#### 15.1.3.3 Kneaders (Dispersers, Masticators)

A common design of kneader consists of a horizontal trough with a saddle-shaped bottom. Two heavy blades mounted on parallel, horizontal shafts rotate towards each other at the top of their cycle. The blades draw the mass of material down over the point of the saddle and then shear it between the blades and the wall and bottom of the trough. The blades may move tangentially to each other and often at different speeds, with a ratio of 1.5:1.0. In some such mixers the blades may overlap and turn at the same or different speeds. Mixing times are generally in the range 2–20 min. One type of kneader, featuring Z- or  $\Sigma$ -blade mixing elements is shown in Fig. 15.7.

#### 15.1.3.4 Continuous Mixers for Pastelike Materials

Many different devices are used to mix viscous materials on a continuous basis. Screw conveyors, rotating inside barrels, may force such materials through perforated plates or wire meshes. Passing them between rollers can effect mixing of pastelike materials. Colloid mills may be used for a similar purpose, see Section 15.2.3.7.

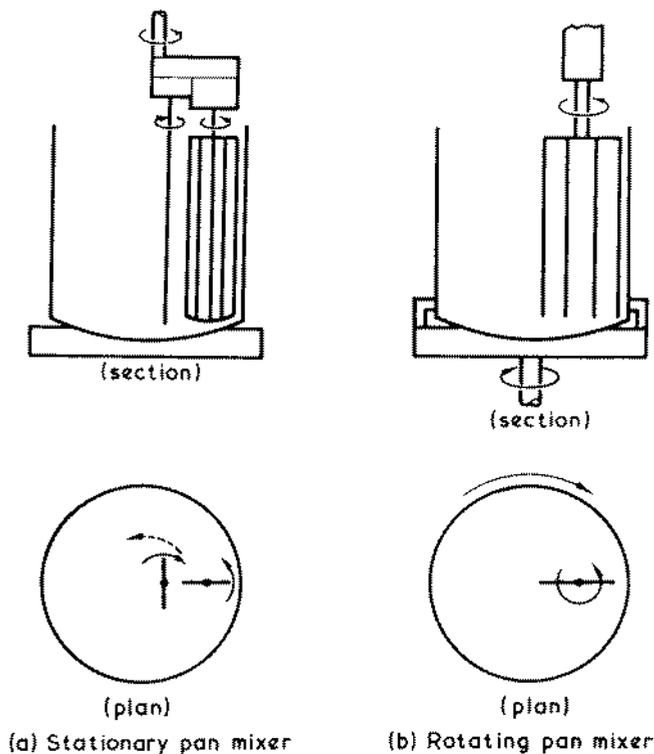


Fig. 15.6 Pan mixers; from [6] with permission of the authors.

#### 15.1.3.5 Static Inline Mixers

When viscous liquids are pumped over specially shaped stationary mixing elements located in pipes, mixing may occur. The liquids are split and made to flow in various different directions, depending on the design of the mixing elements. Many different configurations are available. The energy required to pump the materials through these mixing elements is usually less than that required to drive the more conventional types of mixers discussed above [1, 5, 6, 8–12].

#### 15.1.4

##### Mixing Dry, Particulate Solids

In most practical mixing operations involving dry particulate solids *unmixing* or *segregation* is likely to occur. Unmixing occurs when particles within a group are free to change their positions. This results in a change in the packing characteristics of the solid particles. Unmixing occurs mainly when particles of different sizes are being mixed. The smaller particles can move through the gaps between the larger particles, leading to segregation. Differences in particle shape



Fig. 15.7 Z-blade ( $\Sigma$ -blade) mixer; by courtesy of Winkworth Machinery Ltd.

and density may also contribute to segregation. Materials with particle sizes of  $75\ \mu\text{m}$  and above are more prone to segregation than those made up of smaller particles. Small cohesive particles, which bind together under the influence of surface forces, do not readily segregate. In mixing operations where segregation occurs, an equilibrium between mixing and unmixing will be established after a certain mixing time. Further mixing is not likely to improve the uniformity of the mix.

There are two basic mechanisms involved in mixing particulate solids, i.e. *convection*, which involves the transfer of masses or groups of particles from one location in the mixer to another, and *diffusion*, which involves the transfer of individual particles from one location to another arising from the distribution of particles over a freshly developed surface. Usually both mechanisms contribute to any mixing operation. However, one mechanism may predominate in a particular type of mixer. Segregation is more likely to occur in mixers in which diffusion predominates.

#### 15.1.4.1 Horizontal Screw and Ribbon Mixers

These consist of horizontal troughs, usually semicylindrical in shape, containing one or two rotating mixing elements. The elements may take the form of single or twin screw conveyors. Alternatively, mixing ribbons may be employed. Two

such ribbons may be mounted on a single rotating shaft. They may be continuous or interrupted. The design is such that one ribbon tends to move the solids in one direction while the other moves them in the opposite direction. If the rate of movement of the particles is the same in both directions, the mixer is operated on a batch principle. If there is a net flow in one direction, the mixer may be operated continuously. The mixing vessel may be jacketed for temperature control. If enclosed, it may be operated under pressure or vacuum. Convection is the predominant mechanism of mixing in this type of mixer. Some segregation may occur but not to a serious extent.

#### 15.1.4.2 Vertical Screw Mixers

These consist of tall, cylindrical or cone-shaped vessels containing a single rotating screw, which elevates and circulates the particles. The screw may be located vertically at the centre of the vessel. Alternatively, it may be set at an angle to the vertical and made to rotate, passing close to the wall of the vessel (Fig. 15.8). The convective mechanism of mixing predominates in such mixers and segregation should not be a serious problem.

#### 15.1.4.3 Tumbling Mixers

These consist of hollow vessels, which rotate about horizontal axes. They are partly filled, up to 50–60% of their volume, with the materials being mixed and then rotated, typically for 5–20 min, to bring about mixing of the contents. Because the main mechanism of mixing is diffusion, segregation can be a problem if the particles vary in size. Various designs are available, some of which are shown in Fig. 15.9. They may contain baffles or stays to enhance the mixing effect and break up agglomerates of particles.

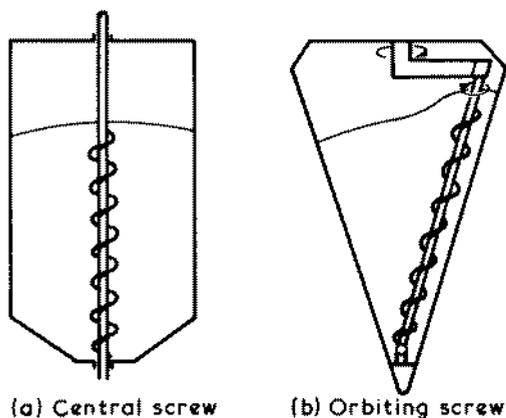


Fig. 15.8 Vertical screw mixers; from [6] with permission of the authors.

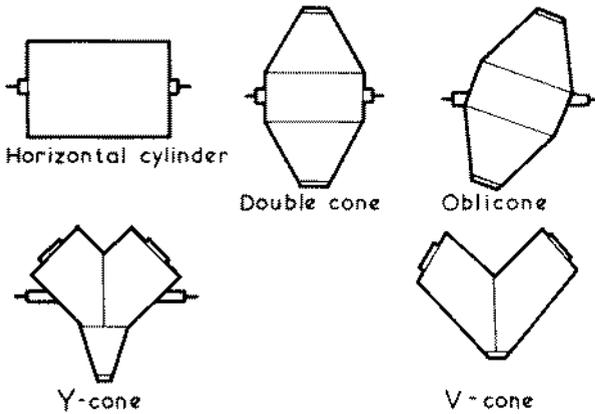


Fig. 15.9 Some typical tumbling mixer shapes; from [6] with permission of the authors.

#### 15.1.4.4 Fluidised Bed Mixers

Fluidised beds, similar to those used in dehydration (see Section 3.2.3.5), may be used for mixing particulate solids with similar sizes, shapes and densities. The inclusion of spouting jets of air, in addition to the main supply of fluidising air, can enhance the mixing [1, 2, 5–8, 13–18].

#### 15.1.5

#### Mixing of Gases and Liquids

Gases may be mixed with low viscosity liquids using impeller agitators in mixing vessels. Turbine agitators, as shown in Fig. 15.3, are generally used for this purpose. In unbaffled vessels, vortexing can draw gas into the liquid. However, the gas may not be well distributed throughout the liquid. It is more usual to use baffled vessels with relatively high-speed turbine impellers. Impellers with six, 12 and even 18 blades have been used. Pitched blades and vaned discs (Fig. 15.3b and d, respectively) are particularly suited to this duty. Some special designs of vaned discs, featuring concave rather than flat blades, have been used for this purpose. Gas may be introduced into liquids using some designs of static inline mixers.

More heavy duty equipment, such as pan and Z-blade mixers, may be used to introduce gas into more viscous materials. For example whisk-like elements in pan mixers may be used to whip creams. Other types of elements, such as forks, hooks and gates, are used to introduce air into doughs and batters. Dynamic, inline systems, such as scraped surface heat exchangers (see Section 2.4.3.3) may be used to heat or cool viscous materials while at the same time introducing gas to aerate them, such as in the manufacture of ice cream, see Section 15.2.4.2 [19].

## 15.1.6

**Applications for Mixing in Food Processing****15.1.6.1 Low Viscosity Liquids**

Examples of applications for impeller mixers include: preparing brines and syrups, preparing liquid sugar mixtures for sweet manufacture, making up fruit squashes, blending oils in the manufacture of margarines and spreads, premixing emulsion ingredients.

**15.1.6.2 Viscous Materials**

Examples of applications for pan mixers and kneaders include: dough and batter mixing in bread, cake and biscuit making, blending of butters, margarines and cooking fats, preparation of processed cheeses and cheese spreads, manufacture of meat and fish pastes.

**15.1.6.3 Particulate Solids**

Examples of application for screw, ribbon and tumbling mixers include: preparing cake and soup mixes, blending of grains prior to milling, blending of flours and incorporation of additives into them.

**15.1.6.4 Gases into Liquids**

Examples of applications of this duty include: carbonation of alcoholic and soft drinks, whipping of dairy and artificial creams, aerating ice cream mix during freezing, supplying gas to fermenters.

**15.2****Emulsification**

## 15.2.1

**Introduction**

Emulsification may be regarded as a mixing operation whereby two or more normally immiscible materials are intimately mixed. Most food emulsions consist of two phases: (a) an aqueous phase consisting of water which may contain salts and sugars in solution and/or other organic and colloidal substances, known as hydrophilic materials and (b) an oil phase which may consist of oils, fats, hydrocarbons, waxes and resins, so called hydrophobic materials. Emulsification is achieved by dispersing one of the phases in the form of droplets or globules throughout the second phase. The material which broken up in this way is known as the dispersed, discontinuous or internal phase, while the other is referred to as the dispersing, continuous or external phase. In addition to the

two main phases other substances, known as *emulsifying agents*, are usually included, in small quantities, to produce stable emulsions.

When the water and oil phases are combined by emulsification, two emulsion structures are possible. The oil phase may become dispersed throughout the aqueous phase to produce an oil in water (o/w) emulsion. Alternatively, the aqueous phase may become dispersed throughout the oil phase to produce a water in oil (w/o) emulsion. In general, an emulsion tends to exhibit the characteristics of the external phase. Two emulsions of similar composition can have different properties, depending on their structure. For example, an o/w emulsion can be diluted with water, coloured with water-soluble dye and has a relatively high electrical conductivity compared to a w/o system. The latter is best diluted with oil and coloured with oil-soluble dye. Many factors can influence the type of emulsion formed when two phases are mixed, including the type of emulsifying agent used, the relative proportions of the phases and the method of preparation employed.

Emulsions with more complex structures, known as *multiple emulsions*, may also be produced. These may have oil in water in oil (o/w/o) or water in oil in water (w/o/w) structures. The latter structure has the most applications. w/o/w emulsions may be produced in two stages. First a w/o emulsion is produced by homogenisation using a hydrophobic agent. This is then incorporated into an aqueous phase using a hydrophilic agent. In such a multiple emulsion, the oil layer is thin and water can permeate through it due to an osmotic pressure gradient between the two aqueous phases. This property is used in certain applications including prolonged drug delivery systems, drug overdose treatments and nutrient administration for special dietary purposes [20].

Emulsions with very small internal phase droplets, in the range 0.0015–0.15  $\mu\text{m}$  in diameter, are termed *microemulsions*. They are clear in appearance, the droplets have a very large contact area and good penetration properties. They have found uses in the application of herbicides and pesticides and the application of drugs both orally and by intravenous injection [21].

In a two-phase system free energy exists at the interface between the two immiscible liquids, due to an imbalance in the cohesive forces of the two materials. Because of this *interfacial tension*, there is a tendency for the interface to contract. Thus, if a crude emulsion is formed by mixing two immiscible liquids, the internal phase will take the form of spherical droplets, representing the smallest surface area per unit volume. If the mixing is stopped, the droplets coalesce to form larger ones and eventually the two phases will completely separate. To form an emulsion, this interfacial tension has to be overcome. Thus, energy must be introduced into the system. This is normally achieved by agitating the liquids. The greater the interfacial tension between the two liquids the more energy is required to disperse the internal phase. Reducing this interfacial tension will facilitate the formation of an emulsion. This is one important role of emulsifying agents. Another function of these agents is to form a protective coating around the droplets of the internal phase and thus prevent them from coalescing and destabilising the emulsion [1, 6, 22, 23].

## 15.2.2

**Emulsifying Agents**

Substances with good emulsifying properties have molecular structures which contain both polar and nonpolar groups. Polar groups have an affinity for water, i.e. are hydrophilic, while nonpolar groups are hydrophobic. If there is a small imbalance between the polar and nonpolar groups in the molecules of a substance, it will be adsorbed at the interface between the phases of an emulsion. These molecules will become aligned at the interface so that the polar groups will point towards the water phase while the nonpolar groups will point towards the oil phase. By bridging the interface in this way, they reduce the interfacial tension between the phases and form a film around the droplets of the internal phase, thus stabilising them. A substance in which the polar group is stronger will be more soluble in water than one in which the nonpolar group predominates. Such a substance will tend to promote the formation of an o/w emulsion. However, if the nonpolar group is dominant the substance will tend to produce a w/o emulsion. If the polar and nonpolar groups are grossly out of balance the substance will be highly soluble in one or other of the phases and will not accumulate at the interface. Such materials do not make good emulsifying agents.

The emulsifying capability of an agent may be classified according to the hydrophile-lipophile balance (HLB) in its molecules. This is defined as the ratio of the weight percentage of hydrophilic groups to the weight percentage of hydrophobic groups in the molecule. HLB values for emulsifying agents range from 1 to 20. Agents with low values, 3–6, promote the formation of w/o emulsions,

**Table 15.1** Examples of emulsifying agents and their HLB values

Ionic	HLB	Nonionic	HLB
Proteins (e.g. gelatin, egg albumin)		Glycerol esters	2.8
Phospholipids (e.g. lecithin)		Polyglycerol esters	
Potassium and sodium salts of oleic acid	18.0–20.0	Propylene glycol fatty acid esters	3.4
Sodium stearoyl-2-lactate		Sorbitol fatty acid esters	4.7
		Polyoxyethylene fatty acid esters	14.9–15.9
Hydrocolloids			
Agar		Carboxymethyl cellulose	
Pectin		Hydroxypropyl cellulose	
Gum tragacanth	11.9	Methyl cellulose	10.5
Alginates		Guar gum	
Carrageen		Locust bean gum	

while those with high values, 8–16, favour the formation of o/w types. Agents with even higher HLB values are used in detergents and solubilisers.

Protein, phospholipids and sterols, which occur naturally in foods, act as emulsifying agents. A wide range of manufactured agents is available, both ionic and nonionic. Ionic emulsifiers often react with other oppositely charged particles such as hydrogen and metallic ions to form complexes. This may result in a reduction in solubility and emulsifying capacity. Nonionic emulsifiers are more widely used in food emulsions. Hydrocolloids such as pectin and gums are often used to increase the viscosity of emulsions to reduce separation of the phases under gravity (creaming). Table 15.1 lists some commonly used emulsifying agents, together with their HLB values. The treatment of the principles of emulsification and the role of emulsifying agents given above is a very simplified one. More detailed accounts will be found in the references cited [6, 22–26].

### 15.2.3

#### **Emulsifying Equipment**

The general principle on which all emulsifying equipment is based is to introduce energy into the system by subjecting the phases to vigorous agitation. The type of agitation which is most effective in this context is that which subjects the large droplets of the internal phase to shear. In this way, these droplets are deformed from their stable spherical shapes and break up into smaller units. If the conditions are suitable and the right type and quantity of emulsifying agent(s) is present, a stable emulsion will be formed. One important condition which influences emulsion formation is temperature. Interfacial tension and viscosity are temperature-dependent, both decreasing with increase in temperature. Thus, raising the temperature of the liquids usually facilitates emulsion formation. However, for any system there will be an upper limit of temperature depending on the heat sensitivity of the components.

#### 15.2.3.1 **Mixers**

In the case of low viscosity liquids, turbine and propeller mixers may be used to pre-mix the phases prior to emulsification. In some simple systems, a stable emulsion may result from such mixing and no further treatment may be required. Special impeller designs are available which promote emulsification, such as the Silverson system shown in Fig. 15.5. In the case of viscous liquids and pastes, pan mixers, kneaders and some types of continuous mixers (see Section 15.1.3) may be used to disperse one phase throughout another. Tumbling mixers, such as those used for mixing powders (see Section 15.1.4.3) may also be used for this purpose.

### 15.2.3.2 Pressure Homogenisers

The principle of operation of all pressure homogenisers is that the premixed phases are pumped through a narrow opening at high velocity. The opening is usually provided between a valve and its seat. Therefore, a pressure homogeniser consists of one or two valves and a high pressure pump. As the liquids pass through the gap, 15–300  $\mu\text{m}$  wide, between the valve and seat they are accelerated to speeds of 50–300  $\text{m s}^{-1}$ . The droplets of the internal phase shear against each other, are distorted and break up into smaller units. As the liquids exit from the gap, there is a sudden drop in pressure. Some cavitation may occur. In many valve designs, the droplets impinge on a hard surface (breaker ring) set at  $90^\circ$  to the direction of flow of the liquids after they emerge from the gap. All of these mechanisms stress the droplets and contribute to their disruption. Droplets diameters of 0.1–0.2  $\mu\text{m}$  are attainable in pressure homogenisers.

There are many different designs of valve available. Three examples are shown in Figure 15.10. As the liquids travel between the valve and its seat, the valve lifts against a heavy duty spring or torsion bar. By adjusting the tension on this spring or bar, the homogenising pressure may be set. This may range from 3.5–70.0  $\text{M N m}^{-2}$ .

The literature suggests that there is an approximately inverse linear relationship between the logarithm of the homogenising pressure and the logarithm of the droplet diameter produced by a pressure homogeniser. Homogeniser valves are usually made of stainless steel or alloys such as stellite. More erosion-resistant materials such as tungsten carbide may be used, but not usually for food applications. It is important that a good fit is maintained between the valves and their seats. Even small amounts of damage to the surfaces can lead to poor



**Fig. 15.10** Three different types of homogenising valve, by courtesy of APV-Rannie & Gaulin Homogenisers.

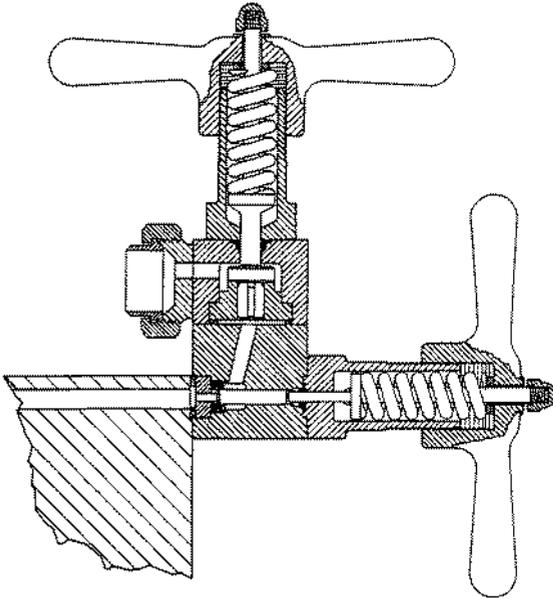


Fig. 15.11 Representation of a two-stage pressure homogeniser.

performance. Consequently, they should be examined regularly and reground or replaced when necessary. Valves made of compressed wire are also available. The liquids are pumped through the myriad of channels in the body of these valves. Such valves are difficult to clean and are discarded at the end of a day's run. They are known as single-service valves.

One passage through a homogenising valve may not produce a well dispersed emulsion. The small droplets of the internal phase may cluster together. These can be dispersed by passing them through a second valve. This is known as two-stage homogenisation. The first valve is set at a high pressure, 14–70 MPa, the second at a relatively low pressure, 2.5–7.0 MPa. A representation of a two-stage homogenising system is shown in Fig. 15.11.

The liquids are pumped through the homogenising valve(s) by means of a positive displacement pump, usually of the piston and cylinder type. In order to achieve a reasonably uniform flow rate, three, five or seven cylinders with pistons are employed, operating consecutively and driven via a crankshaft. The mixture is discharged from the chamber of each piston into a high-pressure manifold and exits from there via the homogenising valve. A pressure gauge is fitted to the manifold to monitor the homogenising pressure.

#### 15.2.3.3 Hydroshear Homogenisers

In this type of homogeniser, the premixed liquids are pumped into a cylindrical chamber at relatively low pressure, up to 2000 kPa. They enter the chamber through a tangential port at its centre and exit via two cone-shaped discharge nozzles at the ends of the chamber. The liquids accelerate to a high velocity as they enter the chamber, spread out to cover the full width of the chamber wall and flow towards the centre, rotating in ever decreasing circles. High shear develops between the adjacent layers of liquid, destabilising the large droplets of the internal phase. In the centre of the cylinder a zone of low pressure develops and cavitation, ultrahigh frequency vibration and shock waves occur which all contribute to the break up of the droplets and the formation of an emulsion. Droplets sizes in the range 2–8  $\mu\text{m}$  are produced by this equipment.

#### 15.2.3.4 Microfluidisers

This type of homogeniser is capable of producing emulsions with very small droplet sizes directly from individual aqueous and oil phases. Separate streams of the aqueous and oil phases are pumped into a chamber under high pressure, up to 110 MPa. The liquids are accelerated to high velocity, impinge on a hard surface and interact with each other. Intense shear and turbulence develop which lead to a break up of the droplets of the internal phase and the formation of an emulsion. Very small emulsion droplets can be produced by recirculating the emulsion a number of times through the microfluidiser.

#### 15.2.3.5 Membrane Homogenisers

If the internal phase liquid is forced to flow through pores in a glass membrane into the external phase liquid an emulsion can be formed. Glass membranes can be manufactured with pores of different diameters to produce emulsions with different droplet sizes, in the range 0.5–10  $\mu\text{m}$ . Such membranes can produce o/w or w/o emulsions with very narrow droplet size distributions. In a batch version of this equipment the internal phase liquid is forced through a cylindrical membrane partly immersed in a vessel containing the external phase. In a continuous version, a cylindrical membrane through which the external phase flows is located within a tube, through which the internal phase flows. The internal phase is put under pressure forcing it through the membrane wall into the external phase. To date, membrane homogenisers are used mainly for the production of emulsions on a laboratory scale.

#### 15.2.3.6 Ultrasonic Homogenisers

When a liquid is subjected to ultrasonic irradiation, alternate cycles of compression and tension develop. This can cause cavitation in any gas bubbles present in the liquid, resulting in the release of energy. This energy can be used to disperse one liquid phase in another to produce an emulsion. For laboratory-scale

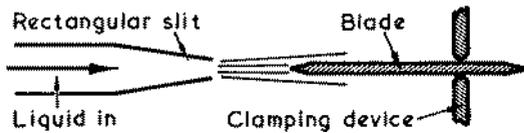


Fig. 15.12 Principle of the mechanical (wedge resonator) ultrasonic generator; from [6] with permission of the authors.

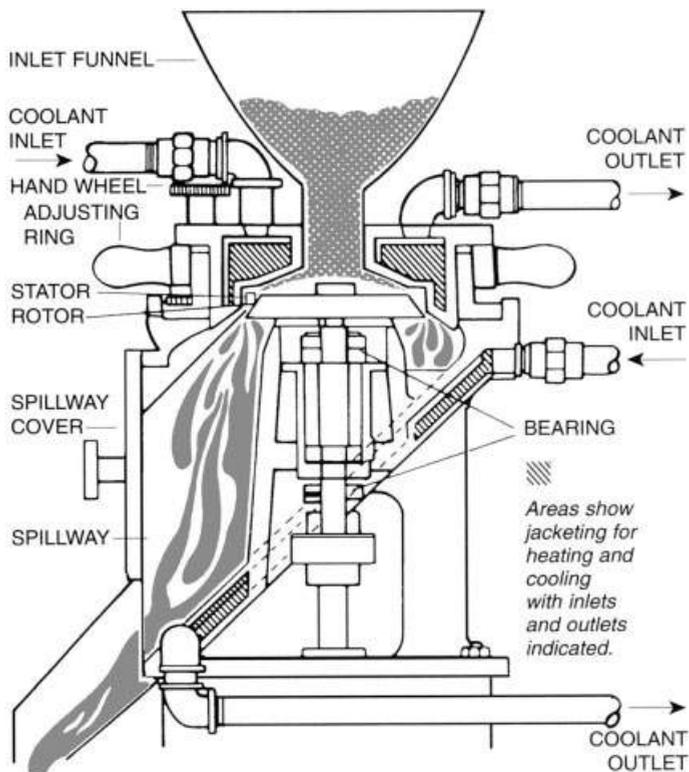
applications, piezoelectric crystal oscillators may be used. An ultrasonic transducer consists of a piezoelectric crystal encased in a metal tube. When a high-intensity electrical wave is applied to such a transducer, the crystal oscillates and generates ultrasonic waves. If a transducer of this type is partly immersed in a vessel containing two liquid phases, together with an appropriate emulsifying agent(s), one phase may be dispersed in the other to produce an emulsion.

For the continuous production of emulsions on an industrial-scale mechanical ultrasonic generators are used. The principle of a wedge resonator (liquid whistle) is shown in Fig. 15.12. A blade with wedge-shaped edges is clamped at one or more nodal points and positioned in front of a nozzle through which the premixed emulsion is pumped. The jet of liquid emerging from the nozzle impinges on the leading edge of the blade, causing it to vibrate at its natural frequency, usually in the range 18–30 kHz. This generates ultrasonic waves in the liquid which cause one phase to become dispersed in the other and the formation of an emulsion. The pumping pressure required is relatively low, usually in the range 350–1500 kPa, and droplet diameters of the order of 1–2  $\mu\text{m}$  are produced.

### 15.2.3.7 Colloid Mills

In a colloid mill, the premixed emulsion ingredients pass through a narrow gap between a stationary surface (stator) and a rotating surface (rotor). In doing so the liquid is subjected to shear and turbulence which brings about further disruption of the droplets of the internal phase and disperses them throughout the external phase. The gap between the stator and rotor is adjustable within the range 50–150  $\mu\text{m}$ . One type of colloid mill is depicted in Fig. 15.13. The rotor turns on a vertical axis in close proximity to the stator. The clearance between them is altered by raising or lowering the stator by means of the adjusting ring. Rotor speed ranges from 3000 rpm for a rotor 25 cm in diameter to 10000 rpm for a smaller rotor 5 cm in diameter. Rotors and stators usually have smooth stainless steel surfaces. Carborundum surfaces are used when milling fibrous materials. Colloid mills are usually jacketed for temperature control. This type of mill, also known as a paste mill, is suitable for emulsifying viscous materials.

For lower viscosity materials the rotor is mounted on a horizontal axis and turns at higher speeds, up to 15000 rpm. Mills fitted with rotors and stators with matching corrugated surfaces are also available. The clearance between the surfaces decreases outwardly from the centre. The product may be discharged



**Fig. 15.13** Top-feed 'paste' colloid mill: by courtesy of Premier Mill Operation, SPX Process Equipment.

under pressure, up to 700 kPa. Incorporation of air into the product is limited and foaming problems reduced in this type of mill [1, 6, 22, 23, 27, 28].

#### 15.2.4

##### Examples of Emulsification in Food Processing

Oil in water (o/w) emulsions of importance in food processing include the following.

##### 15.2.4.1 Milk

This naturally occurring emulsion typically consists of 3.0–4.5% fat dispersed in the form of droplets throughout an aqueous phase which contains sugars and mineral salts in solution and proteins in colloidal suspension. The fat droplets, or globules, range in size from less than 1  $\mu\text{m}$  to more than 20  $\mu\text{m}$  in diameter. These are stabilised by a complex, multilayer coating made up of phospholipids, proteins, enzymes, vitamins and mineral salts, known as the *milk fat globule*

*membrane*. Under the influence of gravity, these globules tend to rise to the surface to form a cream layer when milk is standing in vats or bottles. To prevent such separation milk may be subjected to two-stage homogenisation, reducing the fat globule size to not more than 2 µm in diameter. Pasteurised, homogenised milk is a widely used liquid milk product. Milk and cream which are to be UHT-treated are also homogenised to improve their stability. So also are evaporated milk and cream which are to be heat-sterilised in containers.

#### 15.2.4.2 Ice Cream Mix

This is an o/w emulsion typically containing 10–12% fat dispersed in an aqueous phase containing sugars and organic salts in solution and proteins and some organic salts in colloidal suspension. The stability of the emulsion is important as it has to withstand the rigors of freezing and the incorporation of air to achieve an overrun. In addition to fat, which may be milk fat, vegetable oil or a combination of the two, ice cream mix contains about 10.5% milk solids/nonfat, usually in the form of skim milk powder, and 13% sucrose, dextrose or invert sugar. The milk solids/nonfat acts as an emulsifying agent but additional agents such as esters of mono- and diglycerides are usually included. Stabilisers such as alginates, carrageenan, gums and gelatin are also added. These increase the viscosity of the mix and also have an influence on the proportion of the aqueous phase which crystallises on freezing and the growth of the ice crystals. This in turn affects the texture and melting characteristics of the frozen product. The mix is pasteurised, usually subjected to two-stage pressure homogenisation and aged at 2–5 °C prior to freezing in scraped surface freezers. Some 50% or less of the aqueous phase freezes at this stage and air is incorporated to give an overrun of 60–100%. The product may then be packaged and hardened at temperatures of –20 °C to –40 °C.

#### 15.2.4.3 Cream Liqueurs

These are further examples of o/w dairy emulsions. They need to have longterm stability in an alcoholic environment. Soluble sodium caseinate can be used to stabilise the finely dispersed emulsion.

#### 15.2.4.4 Coffee/Tea Whiteners

These substitutes for cream or milk are also o/w emulsions typically containing vegetable oil, sodium caseinate, corn syrup, high HLB emulsifying agents and potassium phosphate. They are usually prepared by pressure homogenisation and are available in UHT-treated liquid form or in spray dried powder form.

#### 15.2.4.5 Salad Dressings

Many 'French' dressings consist of mixtures of vinegar, oil and various dry ingredients. They are not emulsified as such and the liquid phase separates after mixing. They need to be mixed or shaken thoroughly before use. Other dressings are true o/w emulsions. *Salad cream*, for example, contains typically 30–40% oil, sugar, salt, egg (either yolk or whole egg in liquid or dried form), mustard, herbs, spices, colouring and stabiliser(s). The cream is acidified with vinegar, and/or lemon juice. The lecithin present in the egg usually is the main emulsifying agent, but some additional o/w agents may be added. Gum tragacanth is the stabiliser most commonly used. This increases the viscosity of the emulsion. Being thixotropic, it thins on shaking and facilitates dispensing of the cream. The gum is dispersed in part of the vinegar and water, allowed to stand for up to 4 days until it is fully hydrated and then beaten and sieved. The rest of the aqueous ingredients are premixed, heated to about 80 °C, cooled to about 40 °C and sieved. An emulsion premix is prepared by adding the oil gradually to the aqueous phase with agitation. This premix is then further emulsified by means of a pressure homogeniser, colloid mill or ultrasonic device. It is then vacuum-filled into jars or tubes. *Mayonnaise* is also an o/w emulsion with similar ingredients to salad cream but containing 70–85% oil. The high oil content imparts a high viscosity to the product and stabiliser(s) are usually not required. The premixing is usually carried out at relatively low temperature, 15–20 °C, and a colloid mill used to refine the emulsion.

#### 15.2.4.6 Meat Products

Emulsification of the fat is important in the production of many meat products such as sausages, pastes and pates. Efficient emulsification can prevent fat separation, influence the texture of the product and its behaviour on cooking. Meat emulsions are relatively complex systems. They are usually classed as o/w emulsions but differ in many ways from those discussed above. They are two phase systems consisting of fairly coarse dispersions of solid fat in an aqueous phase containing gelatin, other proteins, minerals and vitamins in solution or colloidal suspension and insoluble matter, including meat fibre, filling materials and seasonings. The emulsifying agents are soluble proteins. Emulsification is brought about simultaneously with size reduction of the insoluble matter in a variety of equipment typified by the mincer and bowl chopper. In the mincer the material is forced by means of a worm through a perforated plate with knives rotating in contact with its surface. It is assumed that some shearing occurs which contributes to the emulsification of the fat. The bowl chopper consists of a hemispherical bowl which rotates slowly about a vertical axis. Curved knives rotate rapidly on a horizontal axis within the bowl. As the bowl rotates it brings the contents into contact with the rotating knives which simultaneously reduce the size of the solid particles and mix the ingredients. Soluble protein is released and emulsification of the fat takes place.

#### 15.2.4.7 Cake Products

*Cake* has been defined as a protein foam stabilised by gelatinised starch and containing fat, sugar, salt, emulsifiers and flavouring materials. It is aerated mainly by gases evolved by chemical reaction involving raising agents. The fat comes from milk, eggs, chocolate and/or added shortenings. The protein comes in the flour, eggs and milk. Emulsifying agents are available in the milk, eggs and flour. Additional agents may be added separately and/or included in the shortenings. Cake batters have an o/w structure. Efficient emulsification of the fat in such batters is essential. Free liquid fat adversely affects the stability of the foam formed on beating and aeration and, consequently, is detrimental to the crumb structure, volume and shape of the baked product. Simultaneous mixing and emulsification is attained in various types of mixers such as pan mixers, operating at relatively high speed, and some continuous mixers. Colloid mills and even pressure homogenisers have been used to ensure good emulsification in low viscosity batters.

Water in oil (w/o) emulsions of importance in food processing include:

#### 15.2.4.8 Butter

This is usually described as a w/o emulsion but, in fact, it has quite a complex structure. The continuous phase of free fat in liquid form contains fat crystals, globular fat, curd granules, crystals of salt, water droplets and gas bubbles. The water droplets remain dispersed due to the semi-solid nature of the continuous phase rather than being stabilised by a layer of emulsifying agent.

Pasteurised milk is separated by centrifugation to give cream containing 30–40% fat. This cream is aged by holding at a low temperature for several hours. The purpose of ageing is to achieve the optimum liquid:solid fat ratio in the butter. Butter may also be made from cultured cream which has been inoculated with lactic acid producing microorganisms and ripened, typically for 20 h at 14 °C, to develop the flavour. In batch churning, the aged cream is tumbled inside a hollow vessel known as a churn. Air is incorporated into the cream and the fat globules concentrate in the surface of the air bubbles. As these break and reform, some of the fat globules break open releasing free fat. The remaining globules form clumps, known as butter grains, held together by some of the free fat. As churning proceeds these grains grow in size. When they reach an optimum size, about 1 cm in diameter, churning is stopped, the aqueous phase, known as buttermilk, is drained off and the grains are washed free of curd with chilled water. The moisture content is adjusted, salt is then added if required and the mass of butter grains is tumbled in the churn for a further period. During this stage of the process, known as working, more fat globules break open, releasing more free fat, water droplets and salt crystals are dispersed throughout the bulk of the fat phase and the texture of the product develops. Working may be carried out under a partial vacuum to reduce the air content of the butter. When working is complete, about 40% of the fat remains in globular form. The butter is discharged from the churn, packaged and transferred to chilled or frozen storage.

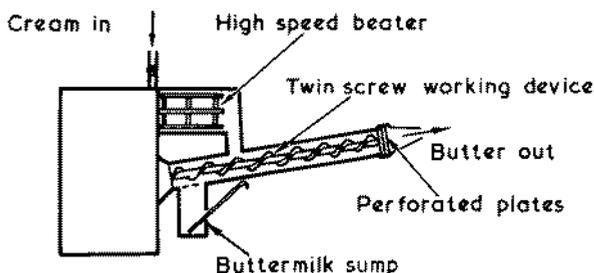


Fig. 15.14 General principle of accelerated churning and working device; from [6] with permission of the authors.

Most continuous methods of buttermaking work on the principles represented in Fig. 15.14. Aged cream containing 30–40% fat is metered into the churning section of the equipment where it is acted on by high-speed beaters. These bring about the rupture of some of the fat globules and the formation of small butter grains. As the grains and buttermilk exit the churning section, the buttermilk drains into a sump and the grains are carried by twin screws up the sloping barrel and extruded through a series of perforated plates. The action of the screws combined with the perforated plates brings about further disruption of fat globules and disperses the remaining water as droplets throughout the fatty phase. The butter is extruded in the form of a continuous ribbon from the working section. Salt may be introduced into the working section, if required.

Other continuous buttermaking methods involve concentrating the fat in the cream to about 80% by a second centrifugation step. This unstable, concentrated, o/w emulsion is then converted into a semi-solid, w/o system by simultaneous agitation and cooling in a variety of equipment.

#### 15.2.4.9 Margarine and Spreads

These w/o products are made from a blend of fats and oils together with cultured milk, emulsifying agents, salt, flavouring compounds and other additives. The blend of fats and oils is selected according to the texture required in the final product. The emulsifying agents used have relatively low HLB values. A typical combination is a mixture of mono- and diglycerides and lecithin. The flavour, originally derived from the cultured milk, is now usually supplemented by the addition of flavouring materials such as acetyl methyl carbinols or aliphatic lactones.

In a typical manufacturing process, the fats and oils are measured into balance tanks. Other ingredients are added and an emulsion premix formed by high-speed agitation. This premix is then pumped through a series of refrigerated, scraped surface heat exchangers where it is simultaneously emulsified and cooled. A three-dimensional network of long, thin, fat crystals is formed. It finally passes through a working/holding device, similar to the working section in Fig. 15.14, where the final texture develops.

Margarine contains 15% water, similar to butter. Low calorie spreads are also made from a blend of oils and contain up to 50% water. They may be based on milk fat or combinations of milk fat and vegetable oils [6, 29–33, 35–38].

## 15.3

### Size Reduction (Crushing, Comminution, Grinding, Milling) of Solids

#### 15.3.1

##### Introduction

Size reduction of solids involves creating smaller mass units from larger mass units of the same material. To bring this about, the larger mass units need to be subjected to stress by the application of force. Three types of force may be applied, i.e. *compression*, *impact* and *shear*. Compressive forces are generally used for the coarse crushing of hard materials. Careful application of compressive forces enables control to be exercised over the breakdown of the material, e.g. to crack open grains of wheat to facilitate separation of the endosperm from the bran (see Section 15.3.3.1). Impact forces are used to mill a wide variety of materials, including fibrous foods. Shear forces are best applied to relatively soft materials, again including fibrous foods. All three types of force are generated in most types of mill, but generally one predominates. For example, in most roller mills compression is the dominant force, impact forces feature strongly in hammer mills and shear forces are dominant in disc attrition mills (see Section 15.3.2).

The extent of the breakdown of a material may be expressed by the *reduction ratio*, which is the average size of the feed particles divided by the average size of the products particles. In this context, the term average size depends on the method of measurement. In the food industry, screening or sieving is widely used to determine particle size distribution in granular materials and powders. In this case, the average diameter of the particles is related to the aperture sizes of the screens used. Size reduction ratios vary from below 8:1 in coarse crushing to more than 100:1 in fine grinding.

The objective in many size reduction operations is to produce particles within a specified size range. Consequently, it is common practice to classify the particles coming from a mill into different size ranges. Again, screening is the technique most widely used for this purpose. To achieve a specified reduction ratio, it may be necessary to carry out the size reduction in a number of stages. A different type of mill may be used in each stage and screens employed between stages. An example of a multistage operation is depicted in Fig. 15.15.

When a solid material is subjected to a force, its behaviour may be represented by a plot of stress versus strain, as shown in Fig. 15.16. Some materials exhibit elastic deformation when the force is first applied. The strain is linearly related to stress (see curve 2 in Fig. 15.16). If the force is removed the solid object returns to its original shape. Elastic deformations are valueless in size re-

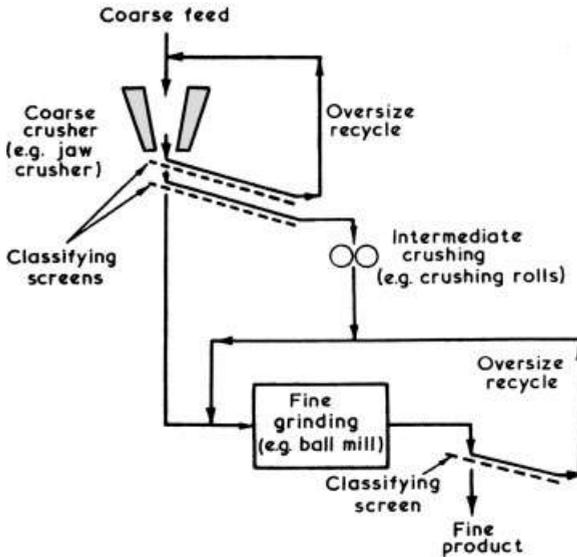
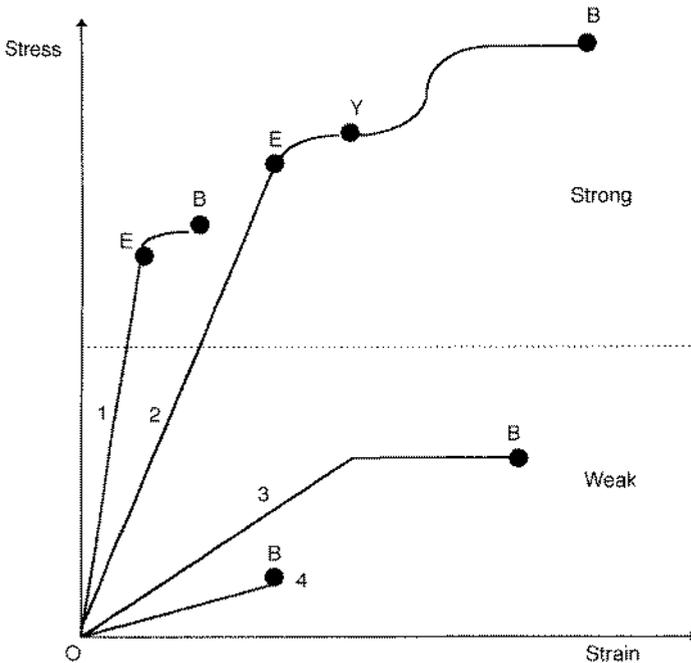


Fig. 15.15 A typical size reduction flow sheet; from [6] with permission of the authors.

duction. Energy is used up but no breakdown occurs. Point *E* is known as the elastic limit. Beyond this point, the material undergoes permanent deformation until it reaches its yield point *Y*. Brittle materials will rupture at this point. Ductile materials will continue to deform, or flow, beyond point *Y* until they reach the break point *B*, when they rupture. The behaviour of different types of material is depicted by the five curves in Fig. 15.16 and explained in the caption to that figure.

The breakdown of friable materials may occur in two stages. Initial fracture may occur along existing fissures or cleavage planes in the body of the material. In the second stage new fissures or crack tips are formed and fracture occurs along these fissures. Larger particles will contain more fissures than smaller ones and hence will fracture more easily. In the case of small particles, new crack tips may need to be created during the milling operation. Thus, the breaking strength of smaller particles is higher than the larger ones. The energy required for particle breakdown increases with decrease in the size of the particles. In the limit of very fine particles, only intermolecular forces must be overcome and further size reduction is very difficult to achieve. However, such very fine grinding is seldom required in food applications.

Only a very small proportion of the energy supplied to a size reduction plant is used in creating new surfaces. Literature values range from 2.0% down to less than 0.1%. Most of the energy is used up by elastic and inelastic deformation of the particles, elastic distortion of the equipment, friction between particles and between particles and the equipment, friction losses in the equipment and the heat, noise and vibration generated by the equipment.



**Fig. 15.16** Stress-strain diagram for various foods: E, elastic limit; Y, yield point; B, breaking point; O-E, elastic region; E-Y, inelastic deformation; Y-B, region of ductility. 1. Hard, strong, brittle material. 2. Hard strong ductile material. 3. Soft, weak, ductile material. 4. Soft, weak, brittle material. Adapted from [39] with permission.

Mathematical models are available to estimate the energy required to bring about a specified reduction in particle size. These are based on the assumption that the energy  $dE$  required to produce a small change  $dx$  in the size of a unit mass of material can be expressed as a power function of the size of the material. Thus:

$$\frac{dE}{dx} = -\frac{K}{x^n} \quad (15.1)$$

*Rittinger's Law* is based on the assumption that the energy required should be proportional to the new surface area produced, i.e.  $n=2$ . So:

$$\frac{dE}{dx} = -\frac{K}{x^2} \quad (15.2)$$

or, integrating:

$$E = K \left[ \frac{1}{x_2} - \frac{1}{x_1} \right] \quad (15.3)$$

Where  $x_1$  is the average initial size of the feed particles,  $x_2$  is the average size of the product particles,  $E$  is the energy per unit mass required to produce this increase in surface area and  $K$  is a constant, known as Rittinger's constant. Rittinger's law has been found to hold better for fine grinding.

*Kick's Law* is based on the assumption that the energy required should be proportional to the size reduction ratio, i.e.  $n = 1$ . So:

$$\frac{dE}{dx} = -\frac{K}{x} \quad (15.4)$$

or, integrating:

$$E = -\ln \frac{x_1}{x_2} \quad (15.5)$$

Where is the size reduction ratio (see above).

Kick's law has been found to apply best to coarse crushing.

In *Bond's Law*,  $n$  is given the value  $3/2$ . So:

$$\frac{dE}{dx} = -\frac{K}{x^{3/2}} \quad (15.6)$$

or, integrating:

$$E = 2K \left[ \frac{1}{(x_2)^{1/2}} - \frac{1}{(x_1)^{1/2}} \right] \quad (15.7)$$

Bond's law has been found to apply well to a variety of materials undergoing coarse, intermediate and fine grinding [1, 6–8, 26, 39, 40].

## 15.3.2

### Size Reduction Equipment

#### 15.3.2.1 Some Factors to Consider When Selecting Size Reduction Equipment

**Mechanical Properties of the Feed** Friable and crystalline materials may fracture easily along cleavage planes. Larger particles will break down more readily than smaller ones. Roller mills are usually employed for such materials. Hard materials, with high moduli of elasticity, may be brittle and fracture rapidly above the elastic limit. Alternatively, they may be ductile and deform extensively before breakdown. Generally, the harder the material, the more difficult it is to break down and the more energy is required. For very hard materials, the dwell time

in the action zone must be extended, which may mean a lower throughput or the use of a relatively large mill. Hard materials are usually abrasive and so the working surfaces should be made of hard wearing material, such as manganese steel, and should be easy to remove and replace. Such mills are relatively slow moving and need to be of robust construction. Tough materials have the ability to resist the propagation of cracks and are difficult to breakdown. Fibres tend to increase toughness by relieving stress concentrations at the ends of the cracks. Disc mills, pin-disc mills or cutting devices are used to break down fibrous materials.

**Moisture Content of the Feed** The moisture content of the feed can be of importance in milling. If it is too high, the efficiency and throughput of a mill and the free flowing characteristics of the product may be adversely affected. In some cases, if the feed material is too dry, it may not breakdown in an appropriate way. For example, if the moisture content of wheat grains are too high, they may deform rather than crack open to release the endosperm. Or, if they are too dry, the bran may break up into fine particles which may not be separated by the screens and may contaminate the white flour. Each type of grain will have an optimum moisture content for milling. Wheat is usually 'conditioned' to the optimum moisture content before milling (see Section 15.3.3.1). Another problem in milling very dry materials is the formation of dust, which can cause respiratory problems in operatives and is a fire and explosion hazard.

In *wet milling*, the feed materials is carried through the action zone of the mill in a stream of water.

**Temperature Sensitivity of the Feed** A considerable amount of heat may be generated in a mill, particularly if it operates at high speed. This arises from friction and particles being stressed within their elastic limits. This heat can cause the temperature of the feed to rise significantly and a loss in quality could result. If the softening or melting temperatures of the materials are exceeded the performance of the mill may be impaired. Some mills are equipped with cooling jackets to reduce these effects.

*Cryogenic milling* involves mixing solid carbon dioxide or liquid nitrogen with the feed. This reduces undesirable heating effects. It can also facilitate the milling of fibrous materials, such as meats, into fine particles.

#### 15.3.2.2 Roller Mills (Crushing Rolls)

A common type of roller mill consists of two cylindrical steel rolls, mounted on horizontal axes and rotating towards each other. The particles of feed are directed between the rollers from above. They are nipped and pulled through the rolls where they are subjected to compressive forces, which bring about their breakdown. If the rolls turn at different speeds shear forces may be generated which will also contribute to the breakdown of the feed particles. The roll surfaces may be smooth, corrugated, grooved, fluted or they may have intermeshing

teeth or lugs. In food applications smooth, grooved or fluted rolls are most often used. Large rolls, with diameters greater than 500 mm, rotate at speeds in the range 50–300 rpm. Smaller rolls may turn at higher speeds. Usually the clearance between the rolls, the *nip*, is adjustable. An overload compression spring is usually fitted to protect the roll surfaces from damage should a hard object try to pass between them (see Fig. 15.17).

The surface of the rolls may be cooled or heated by circulating water or some other thermal fluid within their interior. Large, smooth surfaced rolls are used for relatively coarse crushing, usually achieving a reduction ratio of 4 or lower. Smaller rolls, with different surface configurations, operating at higher speeds can achieve higher ratios. Smooth or fluted rolls, operating at the same or slightly different speeds, are used to crack open grains and seeds. For finer milling, shallow grooves and larger differential speeds are employed. For very fine milling, smooth surfaced rolls, operating at high differential speeds are used. To achieve high reduction ratios, the material being milled may be made to pass between two or more pairs of rolls in sequence, with the clearance decreasing from one pair to the next. Some separation of the particles into different size ranges may take place between each pair of rolls. This principle is employed in the milling of wheat grains (see Section 15.3.3.1). Machines consisting of two, three or more smooth-surfaced rolls, arranged in either a horizontal or vertical sequence, are used to mill liquid products. The product passes between the rolls in a zigzag flow pattern. The clearance between the rolls decreases in the direction of flow of the product and the speeds of consecutive rolls differ, generating shear forces.

Instead of two rolls operating against each other, one roll may operate against a flat or curved hard surface. An example is the *edge mill* (see Fig. 15.18). The heavy rolls roll and slip over a table. Usually the rolls are driven, but in some machines the table is driven instead. The rolls and table surfaces may be smooth or grooved.

In the case of a mill consisting of two rolls rotating towards each other, the *angle of nip*,  $\alpha$ , is the term used to describe the angle formed by the tangents to

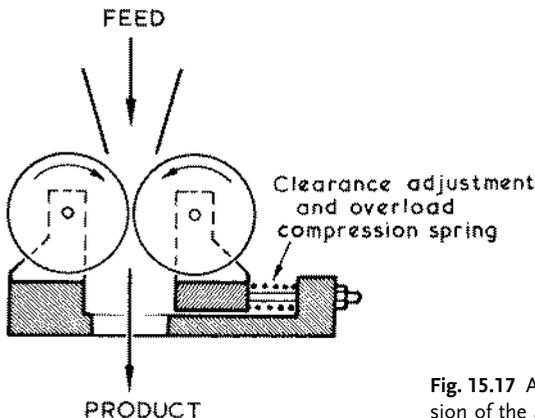


Fig. 15.17 A roller mill; from [6] with permission of the authors.

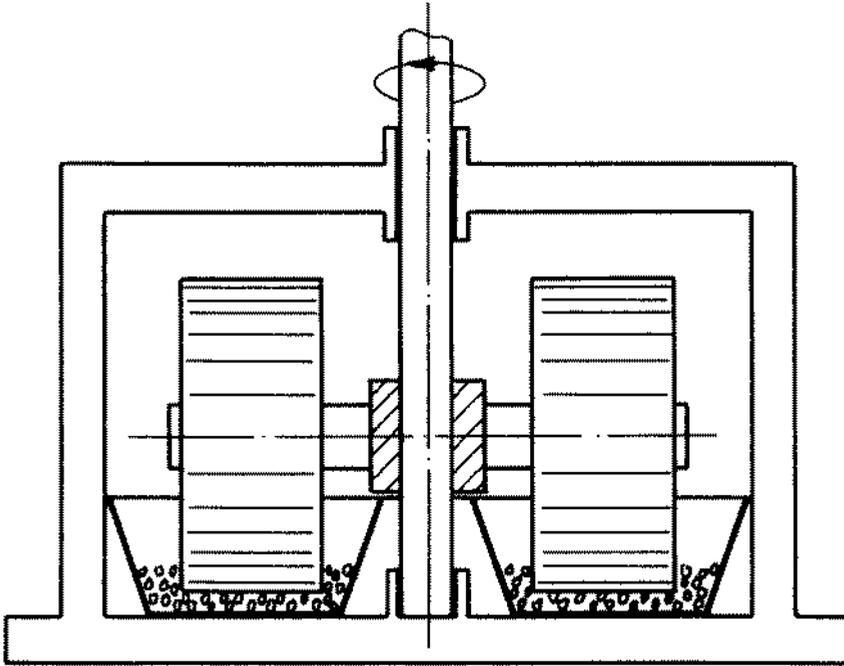


Fig. 15.18 Edge mill; from [8] with kind permission of Springer Science and Business Media.

the roll faces at the point of contact between the particle and the rolls. It can be shown that:

$$\cos \frac{\alpha}{2} = \frac{D_r + D_p}{D_r + D_f} \quad (15.8)$$

Where  $D_f$  is the average diameter of the feed particles,  $D_p$  is the average diameter of the product particles, corresponding to the clearance between the rolls, and  $D_r$  is the diameter of the rolls.

For the particle to be 'nipped' down through the rolls by friction:

$$\tan \frac{\alpha}{2} = \mu \quad (15.9)$$

Where  $\mu$  is the coefficient of friction between the particle and the rolls.

Equations (15.8) and (15.9) can be used to estimate the largest size of feed particle that a pair of rolls will accommodate.

The theoretical mass flow rate  $M$  ( $\text{kg s}^{-1}$ ) of product from a pair of rolls of diameter  $D_r$  (m) and length  $l$  (m) when the clearance between the rolls is  $D_p$  (m), the roll speed is  $N$  (rpm) and the bulk density of the product is  $\rho$  ( $\text{kg m}^{-3}$ ) is given by:

$$M = \frac{\pi D_r N D_p l \rho}{60} \quad (15.10)$$

The literature suggests that the actual mass flow rate is usually between 0.1 and 0.3 of the theoretical value [6–8, 39–41].

### 15.3.2.3 Impact (Percussion) Mills

When two bodies collide, i.e. impact, they compress each other until they have the same velocity and remain in this state until restitution of the compression begins. Then the bodies push each other apart and separate. If one of the bodies is held in position, the other body has to conform with this position for a short interval of time. During the very short time it takes for restitution of compression to occur, a body possesses strain energy which can lead to fracture. The faster the bodies move away from each other the more energy is available to bring about fracture. The faster the rate at which the force is applied the more quickly fracture is likely to occur [40].

**Hammer Mill** In this type of mill, a rotor mounted on an horizontal shaft turns at high speed inside a casing. The rotor carries hammers which pass within a small clearance of the casing. The hammers may be hinged to the shaft so that heads swing out as it rotates. In some designs, the hammers are attached to the shaft by rigid connections. A toughened plate, known as the breaker plate, may be fitted inside the casing, see Fig. 15.19. The hammers and breaker plate are made of hard wearing materials such as manganese steel. The hammers drive the feed particles against the breaker plate. Fracture of the particles is brought about mainly by impact forces, but shear forces also play a part. The casing may be fitted with a screen through which the product is discharged. The size of the screen aperture determines the upper limit particle size in the product. This way of operating a mill is known as *choke feeding*. When milling friable materials, choke feeding may result in a high proportion of very small particles in the product. When milling fibrous or sticky materials, the screen may become blocked. In some such cases the screen may be removed. The mill casing may be equipped with a cooling jacket. The hammer mill is a general purpose mill used for hard, friable, fibrous and sticky materials.

**Beater Bar Mill** In this type of mill, the hammers are replaced by bars in the form of a cross. The tips of the bars pass within a small clearance of the casing. Beater bars are mainly used in small machines.

**Comminuting Mill** Knives replace the hammers or bars in this type of mill. They may be hinged to the shaft so that the swing out as it rotates. Alternatively, they may be rigidly fixed to the shaft. Such mills are used for comminuting relatively soft materials, such as fruit and vegetable matter. In some designs, the knives are sharp on one edge and blunt on the other. When the shaft rotates

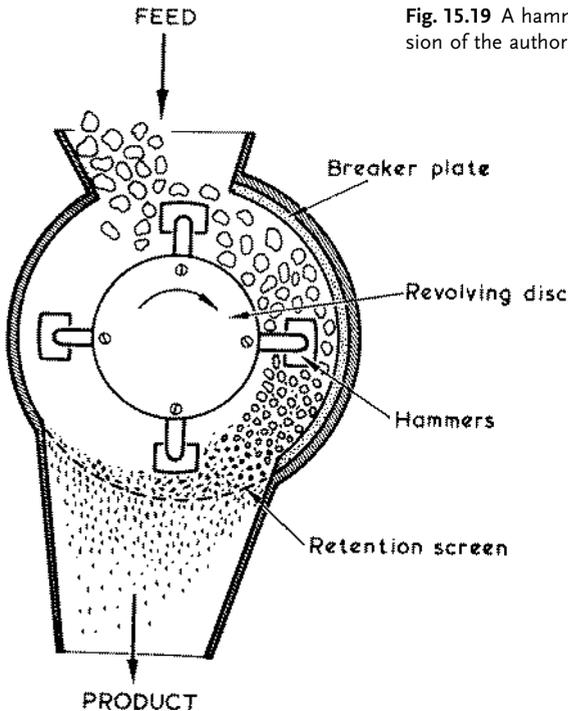
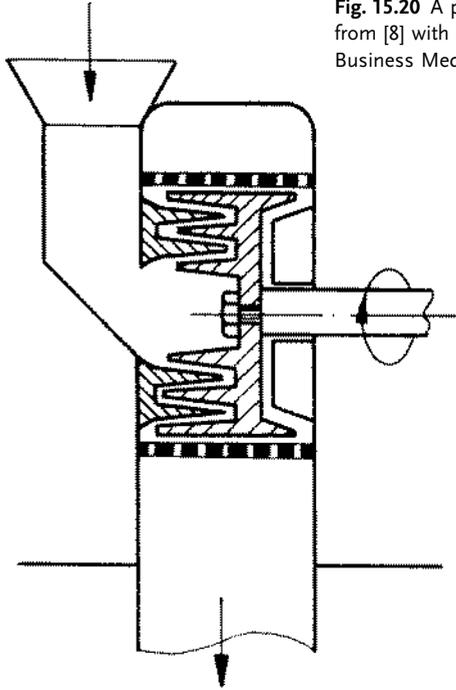


Fig. 15.19 A hammer mill; from [6] with permission of the authors.

in one direction the machine has a cutting action. When the direction of rotation of the shaft is reversed, the blunt edges of the knives act as beater bars.

**Pin (Pin-Disc) Mill** In one type of pin mill a stationary disc and rotating disc are located facing each other, separated by a small clearance. Both discs have concentric rows of pins, pegs or teeth. The rows of one disc fit alternately into the rows of the other disc. The pins may be of different shapes; round, square or in the form of blades. The feed is introduced through the centre of the stationary disc and passes radially outwards through the mill where it is subjected to impact and shear forces between the stationary and rotating pins. The mill may be operated in a choke feed mode by having a screen fitted over the whole or part of the periphery (see Fig. 15.20). Alternatively, it may not have a screen and the material is carried through the mill by an air stream (see Fig. 15.21). In another type of mill, both discs rotate either in the same direction at different speeds or in opposite directions. In some mills the clearance between the discs is adjustable. Disc speeds may be up to 10000 rpm. Pin mills may be fitted with jackets for temperature control. Such mills are suitable for fine grinding friable materials and for breaking down fibrous substances.

**Fluid Energy (Jet) Mill** In this type of mill, the solid particles to be comminuted are suspended in a gas stream travelling at high velocity into a grinding cham-



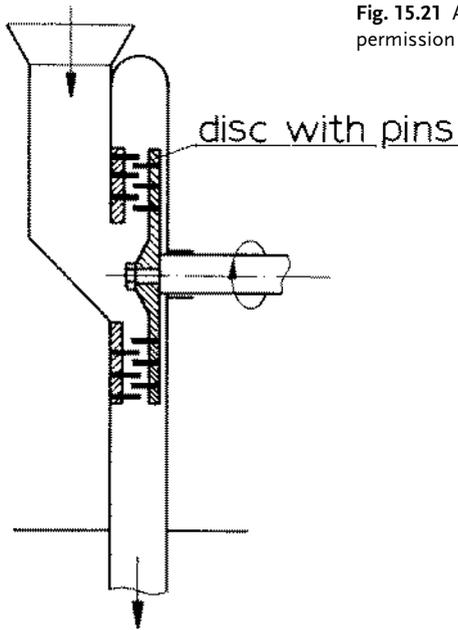
**Fig. 15.20** A pin mill fitted with screen over the periphery; from [8] with kind permission of Springer Science and Business Media.

ber. Breakdown occurs through the impact between individual particles and with the wall of the chamber. The gases used are compressed air or superheated steam, which are admitted to the chamber at a pressure of the order of 700 kPa. An air-solids separation system, usually a cyclone, is used to recover the product. Particles up to 10 mm can be handled in these mills but usually the feed consists of particles less than 150  $\mu\text{m}$ . The product has a relatively narrow size range. Since there are no moving parts or grinding media involved, product contamination and maintenance costs are relatively low. However, the energy efficiency of such mills is relatively low [6–8, 39–41].

#### 15.3.2.4 Attrition Mills

The principle of attrition mills is that the material is rubbed between two surfaces. Both pressure and frictional forces are generated. The extent to which either of these forces predominates depends on the pressure with which both surface are held together and the difference in the speed of rotation of the surfaces.

**Buhrstone Mill** This is the oldest form of attrition mill. It consists of two stones, one located above the other. The upper stone is usually stationary while the bottom one rotates. Matching grooves are cut in the stones and these make



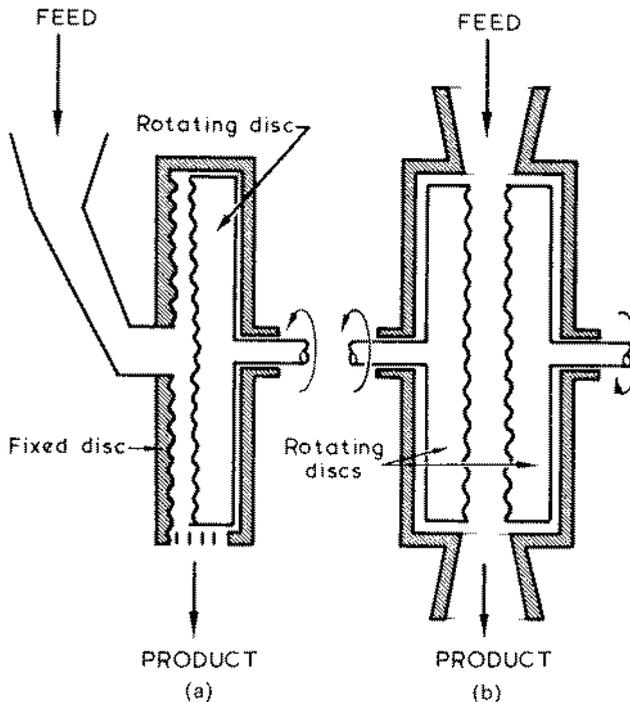
**Fig. 15.21** A pin mill without screen; from [8] with kind permission of Springer Science and Business Media.

a scissor action as the lower one rotates. The feed is introduced through a hole in the centre of the upper stone and gradually moves outwards between the stones and is discharged over the edge of the lower stone. In some such mills both stones rotate, in opposite directions. Siliceous stone is used. In more recent times toughened steel 'stones' have replaced the natural stone for some applications. Such stone mills were used for milling of wheat for centuries and are still used to produce wholegrain flour today. They are often driven by wind-mills. They are also used for wet milling of corn.

**Single-Disc Attrition Mill** In this type of mill a grooved disc rotates in close proximity to a stationary disc with matching grooves. The feed is introduced through the centre of the stationary disc and makes its way outwards between the discs and is discharged from the mill via a screen (see Fig. 15.22a). Shear forces are mainly responsible for the breakdown of the material, but pressure may also play a part. The clearance between the two discs is adjustable.

**Double-Disc Attrition Mill** This attrition mill consists of two counter-rotating discs, with matching grooves, located close to each other in a casing. The feed is introduced from the top and passes between the discs before being discharged, usually through a screen at the bottom of the casing (see Fig. 15.22b). Shear forces, again, predominantly cause the breakdown of the material.

Both types of disc attrition mills are used for milling fibrous materials such as corn and rice.



**Fig. 15.22** Disc mills. (a) A single disc mill. (b) A double disc mill. From [6] with permission of the authors.

**The Foss Mill** This design of mill has studs fitted to the discs instead of grooves. It could be regarded also as a modified pin mill (see Section 15.3.2.3). It is used for similar applications as the other disc mills.

**Colloid Mill** This is another example of an attrition mill which is used for emulsification and in the preparation of pastes and purees (see Section 15.2.3.7) [6–8, 39–41].

#### 15.3.2.5 Tumbling Mills

A typical tumbling mill consists of a cylindrical shell, sometimes with conical ends, which rotates slowly about a horizontal axis and is filled to about 50% of its volume with a solid grinding medium. As the shell rotates the loose units of the grinding medium are lifted up on the rising side of the shell to a certain height. They then cascade and cataract down the surface of the other units. The material being comminuted fills the void spaces between the units of the grinding medium. Size reduction takes place between these units in the jostling as they are lifted up and in the rolling action and impact as they fall down. The most commonly used grinding media are balls and rods. Contamination of the

feed material due to wear of the grinding medium is a problem with tumbling mills and needs to be monitored.

**Ball Mills** In ball mills the grinding medium consists of spherical balls, 25–150 mm in diameter, and usually made of steel. Alternatively, flint pebbles or porcelain or zircon spheres may be used. Mills employing such grinding materials are known as *pebble mills*. At low rotation speeds the balls are not lifted very far up the wall of the cylindrical and tumble over each other as they roll down. In such circumstances, shear forces predominate. At higher speeds, the balls are lifted up higher and some fall back down generating impact forces which contribute to the size reduction of the feed material. Above a certain *critical speed*, the balls will be carried round against the cylinder wall under the influence of centrifugal force and comminution ceases. This critical speed can be calculated from the expression:

$$N = \frac{42.3}{(D)^{1/2}} \quad (15.11)$$

Where  $N$  is the critical speed (rpm) and  $D$  is the diameter of the cylinder (m). Ball mills are run at 65–80% of their critical speeds.

Ball mills may be batch or continuous. In the latter case, the feed material flows steadily through the revolving shell. It enters at one end through a hollow trunnion and leaves at the other end through the trunnion or through peripheral openings in the shell. Ball mills are used for fine grinding and may be operated under dry or wet conditions.

In the *vibration ball mill*, the shell containing the balls is made to vibrate by means of out of balance weights attached to each end of the shaft of a double-ended electric motor. In such mills, impact forces predominate and very fine grinding is attainable.

Another variation of the ball mill, known as an *attritor*, consists of a stationary cylinder filled with balls. A stirrer keeps the balls and feed material in slow motion generating shear and some impact forces. It is best suited to wet milling and may be operated batchwise or continuously. This type of mill is used in chocolate manufacture (see Section 15.3.3.2).

**Rod Mills** Grinding rods, usually made of high carbon steel, are used instead of balls in rod mills. They are 25–125 mm in diameter and may be circular, square or hexagonal in cross-section. They extend to almost the full length of the shell and occupy about 35% of the shell volume. In such mills, attrition forces predominate but impacts also play a part in size reduction. They are classed as intermediate grinders and are more useful than ball mills for milling sticky materials [6–8, 39–41].

## 15.3.3

**Examples of Size Reduction of Solids in Food Processing**15.3.3.1 **Cereals**

**Wheat** The structure of the wheat grain is complex but, in the context of this section, it can be assumed to consist of three parts, i.e. the outer layer or bran, the white starchy endosperm and the embryo or germ. According to Meuser [41]: “The objectives of milling white flour are: 1. To separate the endosperm, which is required for the flour, from the bran and embryo, which are rejected, so that the flour shall be free from bran specks, and of good colour, and so that the palatability and digestibility of the product shall be improved and its storage life lengthened. 2. To reduce the maximum amount of endosperm to flour fineness, thereby obtaining the maximum extraction of white flour from the wheat.” The number of parts of flour by weight produced per 100 parts of wheat is known as the *percentage extraction rate*. The wheat grain contains about 82% of endosperm, but it is not possible to separate all of it from the bran and embryo. In practice, the extraction rate is in the range 70–80%.

Prior to milling, the wheat grains are cleaned to remove metal fragments, stones, animal matter and unwanted vegetable matter and conditioned to the optimum moisture content for milling. A mill consists of two sections, a *break section* and a *reduction section*. The clean grain is fed to the break section, which usually consists of four or five pairs of fluted rolls. The rolls rotate towards each other at different speeds. As the grains pass through the first pair of break rolls they are split open. Some large fragments of endosperm (semolina) are released together with a small amount of small particles, less than 150  $\mu\text{m}$  in size, which is collected as flour. The fragments of bran coming from these rolls will have endosperm attached to them. The fractions from the first break rolls are separated by sieving (see Fig. 15.23). The bran passes through a second set of fluted rolls where more semolina and flour is released. This is repeated two or three more times, until relatively clean bran is collected as a byproduct from the last set of sieves. The clearance between the pairs of break rolls and the depth of the fluting decrease in the direction of flow of the bran. The semolina, which contains the germ and some particles, goes to the reduction section. This section consists of up to 16 pairs of smooth surfaced rolls, rotating towards each other at different speeds. The speed differential is less than that between the break rolls. As the semolina passes through the first set of reduction rolls, some of the large fragments are broken down into flour; and the germ, which is relatively soft, is pressed into flakes. These fractions are separated by sieving. The germ is usually discharged from this set of sieves as another byproduct. The large particles of semolina pass through another set of reduction rolls where more flour is produced. This is repeated up to fourteen more times. In addition to some flour, another byproduct, consisting mainly of fine bran particles and known as wheat feed, is discharged from the last set of sieves. The clearance between the pairs of reduction rolls decreases in the direction of flow of the semolina. The greatest proportion of flour is collected from the early reduc-

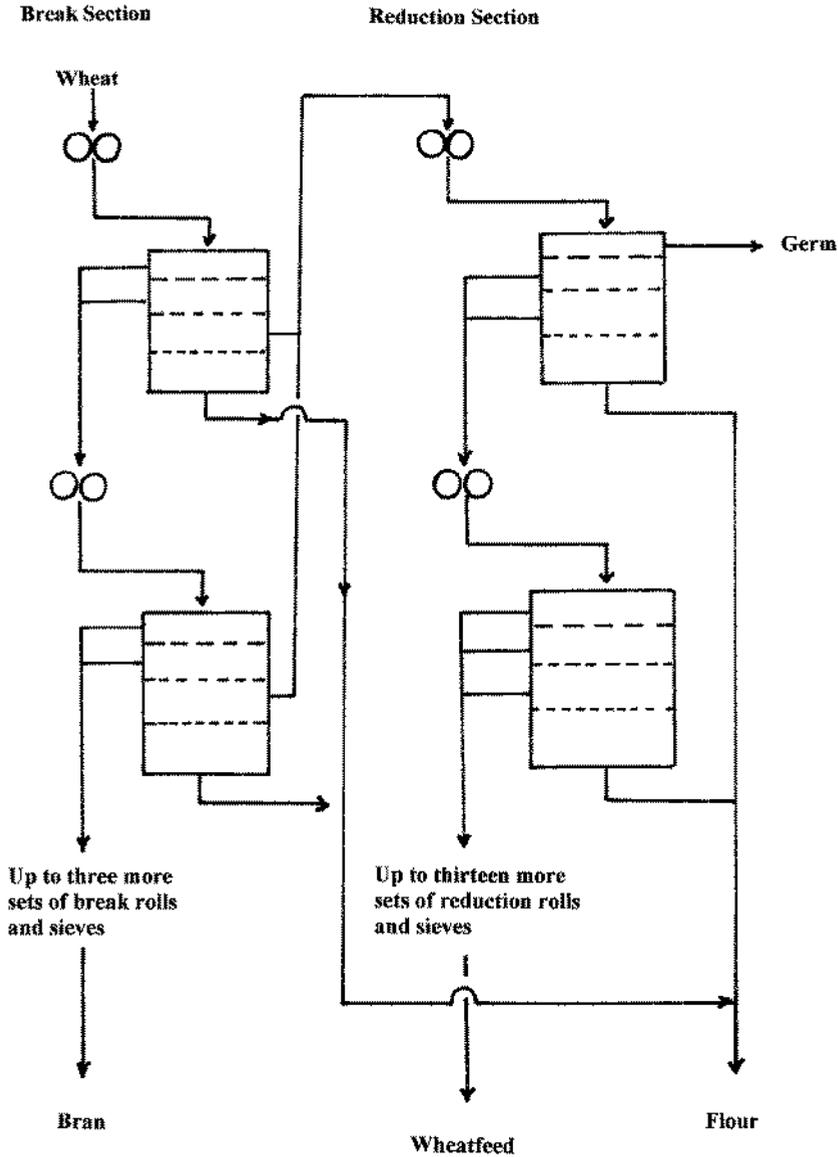


Fig. 15.23 The principle of a wheat milling process.

tion rolls. The flour coming from the later reduction rolls contains more fine bran particles and is darker in colour than that collected from the early rolls.

This is a rather simplified description of white flour milling. More detailed accounts are available in the literature [41, 42]. Recent developments in flour milling are discussed in [43]. Wholemeal flour contains all the products of

milling cleaned wheat. It is produced using millstones (see Section 15.3.2.4), by roller mills using a shortened system or by attrition mills. Brown flours, which have extraction rates of 85–98%, are produced by a modified white flour milling system. The wheat may be conditioned to a lower moisture content than that used for white flour so that the bran breaks into smaller particles. Durum wheat is milled to produce coarsely ground endosperm, known as semolina, which is used in the manufacture of pasta and as couscous.

Sorghum, millets and maize and rye may be dry milled using modified flour milling systems.

**Rice** Unlike most milled cereal products, in the production of milled rice the endosperm is kept as intact as possible. Brown rice is dehulled using rubber covered rolls or by means of abrasive discs. The bran and embryo are then removed, a process known as whitening. This may be achieved by means of abrasive cones or rolls. A further step, known as polishing, removes the aleurone layer, which is rich in oil. This extends the storage life of the rice by reducing the tendency for oxidative rancidity to occur. Polishing is achieved by means of a rotating cone covered with leather strips.

**Maize** This cereal is wet milled to produce a range of products including starch, oil and various types of cattle feed. Cleaned maize is steeped in water, containing 0.1–0.2% sulphur dioxide, at 50 °C for 28–48 h. The steeping softens the kernel and facilitates separation of the hull, germ and fibre from each other. The SO<sub>2</sub> may disrupt the -SS- bonds in the protein, enabling starch/protein separation. After steeping, the steep water is concentrated by vacuum evaporation and the protein it contains is recovered by settling. The steeped maize is coarsely milled in a Foos Mill (see Section 15.3.2.4). The grain is cracked open and the germ released. The germ is recovered by settling or by means of hydrocyclones. Oil may be extracted from the germ by pressing. The degermed material is strained off from the liquid and milled in an impact or attrition mill. The hulls and fibre are separated from the protein and starch by screening. The suspension of starch and protein coming from the screen is fed to high-speed centrifuges where they are separated from each other. The starch is purified in hydrocyclones, filtered and dried. The protein is also filtered and dried. The products from the wet milling of maize include about 66% starch, 4% oil and 30% animal feed. Most of the starch is converted into modified starches, sweeteners, alcohol and other useful products [42–46].

### 15.3.3.2 **Chocolate**

Size reduction equipment is employed at several stages in the manufacture of chocolate. The first stage is breaking the shell of the cocoa bean to release the nib. The beans are predried in heated air, or steam puffed, or exposed to infrared radiation to weaken the shell and loosen the nib. The bean shells are generally broken by a type of impact mill. The beans are fed onto plate or disc, rotat-

ing at high speed. They are flung against breaker plates by centrifugal force. Sometimes they fall onto a second rotating disc, which flings them back against the plate again. The size distribution of the product from the mill should be as narrow as possible, ideally with the size range 7.0–1.5 mm. The nibs are separated from the fragments of shell by a combination of sieving and air classification (winnowing). It is important that this separation is as complete as possible, as the presence of shell can cause excessive wear on the equipment used subsequently to grind the nibs and refine the chocolate. The next application for size reduction equipment is the grinding of cocoa nibs. These are ground to cocoa liquor for the removal of cocoa butter from within the cellular structure. Cocoa nib contains about 55% butter, contained within cells which are 20–30  $\mu\text{m}$  in size. These cells must be broken to release the butter. The cell wall material and any shell and germ present are fibrous and tough. The particle size range after grinding should be in the range 15–50  $\mu\text{m}$ . The grinding of the nibs is usually carried out in two steps. They are first preground in a hammer, disc or pin mill. The second step is carried out in an agitated ball mill or attrition mill. The first of these mills consists of a cylinder filled to as much as 90% of its volume with balls made of steel or ceramic material. For large throughputs, a series of ball mills may be used, with the size of the balls decreasing in the direction of flow of the liquor, from 15 mm down to 2 mm. Some contamination of the liquor is to be expected due to wear of the equipment. Three single-disc attrition mills, working in series, may replace the ball mill. The three pairs of discs may be housed in the one machine. These can be fed directly with dry nibs, but pregrinding is more usual.

A further stage in chocolate manufacture is refining. The purpose of refining is to ensure that the particle size of the dispersed phase is sufficiently small so as not to impart a gritty texture to the chocolate when it is eaten. Usually this means that the largest particles should be in the range 15–30  $\mu\text{m}$ . If the cocoa liquor has been properly ground, the purpose of refining is to reduce the size of the added sugar crystals and, in the case of milk chocolate, the particles of milk powder. Refining is usually carried out using a five-roll machine. The gap between the rolls decreases from bottom to top while the speed of rotation increases. The cocoa liquor is introduced between the bottom two rolls. The film of chocolate leaving each gap is transferred to the faster roll and so moves upwards. The product is scraped off the top roll.

Uncompressed rolls are barrel-shaped. Hydraulic pressure is applied to the roll stack to compress the camber on the rolls and obtain an even coating across the length of each roll. The reduction ratio attainable is 5–10, which should result in the particles in the product being in the correct size range. The temperature of the rolls is controlled by circulation of water through them. It is important that the feed material to the rolls is well mixed. In some systems, a two-roll refiner known as a prerefiner is employed to prepare the feed for the main roll stack.

Cocoa powder is produced by partially defatting ground cocoa nibs in a press. The broken cake from the press is blended prior to milling to standardise the colour. The blend is fed to a pin mill where it is broken down to a powder. The

powder is cooled to about 18°C by being conveyed through jacketed tubes by compressed air. At the end of the cooling line, the powder is recovered from the air by means of a cyclone. It is transported to a stabiliser, where the latent heat is removed to solidify the remaining fat [47–49].

#### 15.3.3.3 Coffee Beans

After roasting, coffee beans are cooled and then ground. The extent of the grinding depends on the how the coffee is to be brewed. Generally, coffee which is to be percolated will have a larger particles size than filter coffee. The actual particle sizes vary from country to country. In Europe, coarse ground coffee for percolators has an average particle size of about 850 µm, while that of a medium grind, intended to be filtered, is about 450 µm. Finely ground coffee, about 50 µm, is required for some types of Turkish coffee and for espresso machines. In the latter case, the beans are ground in small amounts just prior to use. Ground beans for large-scale percolation (extraction) for the production of instant coffee may be somewhat coarser than the normal coarse ground product.

The original method of grinding coffee beans was by means of a buhrstone-type mill (see Section 15.3.2.4). These are still in use in some developing countries, particularly in markets where the customer grinds his roast beans at the time of purchase. The stones have been replaced by serrated, cast iron discs. For large-scale grinding of coffee beans, multiroll mills are mostly used. Three or four pairs of rolls are located one above the other. In each pair, the rolls rotate at different speeds. The fast roll has U-shaped corrugations running lengthwise while the other has peripheral corrugations. As the beans pass down between successive pairs of rolls, the clearance between them decreases and the roll speed increases. A three-stand mill is used to produce coarse or medium ground coffee. A fourth pair of rolls is used for fine grinding. Chaff remaining after roasting is released during grinding. This may be removed or incorporated with the ground coffee in a type of ribbon mixer. The latter procedure is known as *normalising*. Various types of attrition and hammer mills are used to grind roasted beans on a smaller scale.

A considerable amount of carbon dioxide is release during grinding of roasted beans. Quite high concentrations of the gas can develop in the vicinity of the milling equipment. If ventilation is poor, operatives may be physiologically affected. The main effect is that the person becomes silly and acts oddly, without realising why. Water vapour and volatile flavour compounds are also released during grinding. Cryogenic grinding, using solid carbon dioxide or liquid nitrogen, may be employed to maximise the retention of the flavour volatiles [50, 51].

#### 15.3.3.4 Oil Seeds and Nuts

It is common practice to flake or grind oilseeds prior to extraction, either by pressing or solvent extraction, in order to rupture the oil cells. Hammer mills and attrition mills are sometimes used for the preliminary breakdown of large

oil seeds such as copra or palm. However, flaking rolls are usually used to prepare seeds for extraction. A common arrangement used for cottonseed, flaxseed and peanuts consists of five rolls located one above the other. The seed is fed in between the top two rolls and then passes back and forth between the rolls down to the bottom of the stack. Each roll supports the weight of the rolls above it, so the seeds are subjected to increasing pressure as they move down the stack. The top roll is grooved, but the others have smooth surfaces [52].

#### 15.3.3.5 Sugar Cane

Large, heavy duty, two- or three-roll crushers are used to break and tear up the cane to prepare it for subsequent milling for the extraction of the sugar. The surfaces of the rolls are grooved to grip the cane and very high pressures are applied to the cane, 8–12 t dm<sup>-2</sup>. Following this initial crushing, the cane may be further broken down in shredding devices, which tear open the cane cells and release some of the juice. These devices are essentially large-capacity hammer mills, with rotors turning at 1000–1200 rpm, carrying hammers pivoted to discs or plates. The hammers pass very close to an anvil plate made up of rectangular bars. The material leaving the shredders is made up of cell material and long thread-like fibres which hold it together when it is subjected to pressure during extraction of the juice by milling.

To extract the juice, the shredded cane is passed through a series, usually five, of triple-roll mills. These are heavy-duty rolls, with grooved surfaces which exert high pressure, 15–30 t dm<sup>-2</sup> on the cane, expressing the juice. This is not a size reduction operation as such, but is an example of another use of roller mills. Water is added to the material after each set of rolls. Alternatively, the dilute juice from the last mill is sent back before the preceding mill. This procedure, known as *imbibition*, increases the amount of sugar extracted, compared with dry crushing [53].

Numerous other food materials are size-reduced in mills. Mustard seeds are milled in a similar fashion to wheat grains, passing through grooved break rolls and then through smooth-surfaced reduction rolls. Spices are milled using a variety of impact and attrition mills. Considerable heat may be generated and result in a loss of volatile oils and development of undesirable aromas and flavours. Mills may be jacketed and cooled. Cryogenic milling, using liquid nitrogen, can result in higher quality products. Sugar crystals may be ground in impact mills to produce icing sugar. Particles of milk powder, lactose and dry whey may be reduced in size in impact mills.

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