CHAPTER 15

Solids Mixing

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Part A: Fundamentals of Solids Mixing

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15-1 INTRODUCTION

Solids mixing is essential to many industries, including ceramics, metallurgy, chemicals, food, cosmetics, coal, plastics, and pharmaceuticals. To give an idea of the magnitude of applications involving granular processes, worldwide production annually accounts for over a trillion kilograms of granular and powdered products, much of which must be uniformly blended to meet quality and performance goals. In this chapter we present an example-oriented overview of current understanding of mixing and de-mixing mechanisms of importance to powder blending operations. We focus on blending in tumblers, which simultaneously comprises the bulk of solids blending operations and represents the greatest opportunity for future predictive modeling. We direct the reader to existing literature sources (e.g., Harnby, 1997) for more specialized blending equipment.

Numerous distinct mechanisms for both mixing and de-mixing of granular materials have been cataloged, including convection, diffusion, shear, and percolation, and in most applications several mechanisms act concurrently and interact in complex ways. For example, details of loading of powders into blenders of

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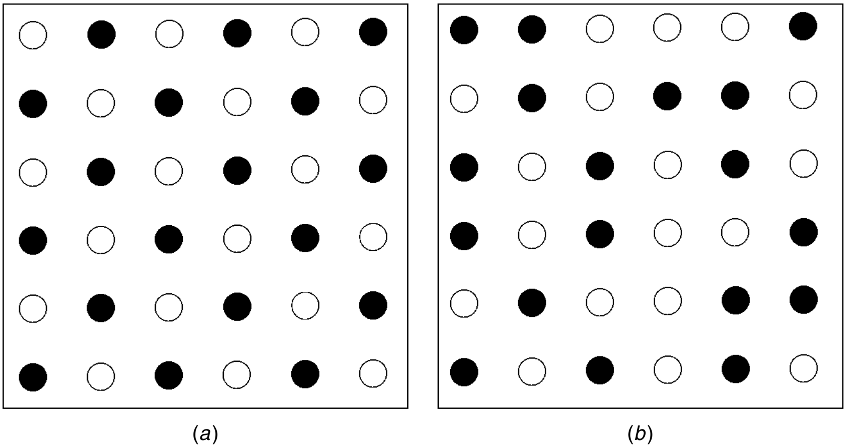
common design can alter the time needed to homogenize them by two orders of magnitude, and by the same token, given that a certain blender can be designed to deliver acceptable performance in the laboratory, we have no consistent a priori mechanism to scale the process up and achieve the same performance in blenders of industrial size. The opposite problem, lack of dynamical similarity during process scale-down, is also quite common, haunting practitioners who attempt to undertake benchtop product design or wish to reproduce manufacturing problems in the lab. Nevertheless, although comprehensive predictive understanding of practical blending problems remains a distant goal, it has recently become possible to define models that generate respectable agreement with observations in practical granular devices (e.g., 3D tumblers). Progress has been made to develop systematic techniques to analyze new products and equipment. Some of these advances are reviewed in this chapter, following a description of the current level of understanding of blending and segregation mechanisms in commonly used industrial devices.

15-2 CHARACTERIZATION OF POWDER MIXTURES

A prerequisite to meaningful evaluation and interpretation of mixing is the development of a reliable measure of mixing. Straightforward though this concept may seem, some care needs to be exercised in its implementation. Any mixing measure is obtained by first evaluating a relevant quantity, typically concentration, in specified sample regions. Ideally, for the samples to be representative, they should be taken uniformly from a flowing stream that is itself uniform in both space and time. In tumbling blenders, this is not practical, and sampling usually consists of extracting small samples from a static bed. We discuss techniques for extracting such samples shortly, but first it is worthwhile to review the description of ideal mixtures, for which particle distributions are known throughout the mixture.

15-2.1 Ideal Mixtures versus Real Mixtures

Mixing is so common an every day experience to both specialist and layperson that it is often taken for granted. Throughout the undergraduate curriculum in engineering, processes that are clearly mixing-dependent (such as chemical reaction, crystallization, die filling) are assumed to be homogeneous. This widespread preconception is also reflected in the common attitude toward powder mixtures, especially for relatively small particles that, due to their ability to scatter visible light, tend to look more uniform to the naked eye than is often warranted. Early conceptions of homogeneous particle assemblies assumed the particles to be distributed in a state of perfect homogeneity, such that any sample containing a large number of particles would have essentially the same composition. Three conceptual approaches to such blissful state—perfect, random, and ordered mixtures—are discussed below. Real mixtures, unfortunately, tend to show at least some degree of heterogeneity, obeying to one of three main causes: incomplete



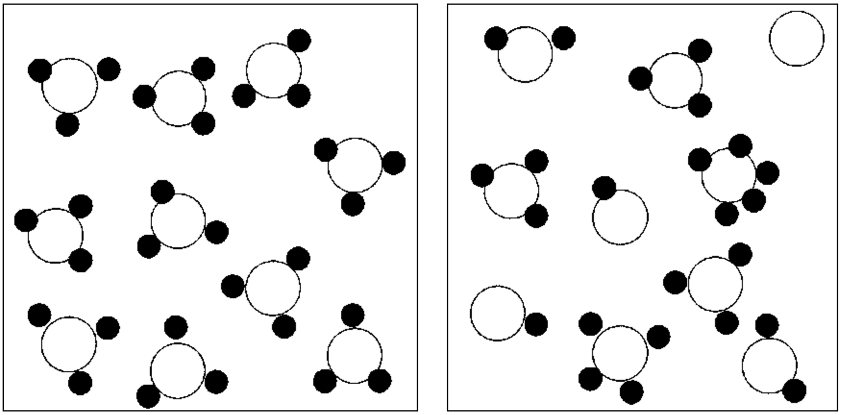
(*a*) (*b*)

Figure 15-1 Simulated mixtures: (*a*) perfect mixture; (*b*) random mixture. (From Williams, 1986.)

mixing, agglomeration, and segregation, resulting in different types of textures, also discussed below.

15-2.1.1 Perfect Mixtures versus Random Mixtures. The first and simplest conception of a homogeneous system is the perfectly uniform mixture, where particles alternate themselves along a lattice (Figure 15-1*a*), very much resembling the position of atoms of different species inside a perfect crystal. Samples taken from such a mixture are necessarily identical. This highly ordered state is never achieved unless painstakingly created by positioning particles one at a time. If the particles are freely moving and differing from one another by a property that does not affect their movement in any way (such as, perhaps, color for identically sized glass beads), the best achievable state is that of a random mixture (Figure 15-1*b*), rigorously defined as a mixture where the probability of a particle belonging to a certain moiety is statistically independent of the nature of its neighbors. Sample extracted from such a mixture follow a binomial (or multinomial) distribution.

15-2.1.2 Ordered Mixtures. For cohesive systems where the particles apply surface forces to one another, it is common to observe the formation of agglomerates. Depending on the relative magnitude of forces between like-particles and unlike-particles, it is possible to see agglomerates of a single species (the "guest"), as well as agglomerates where a small-size moiety essentially coats another, larger moiety (the "host"). This latter situation motivated the concept of an "ordered mixture" (which the reader should distinguish from the situation depicted in Figure 15-1*a*). In the ideal case, the same exact number of identical guest particles covers every identical host (Figure 15-2*a*). Samples taken from such a system would be, once again, identical, thus resulting in a higher degree of sample homogeneity than the random mixtures depicted in Figure 15-1*b*. In reality,



(*a*) (*b*)

Figure 15-2 Distributions of individual particles that form an (*a*) ideal random mixture and a (*b*) random mixture. Part (*b*) illustrates a less structured blend that is still well mixed but does not exhibit long-range order in the spatial distribution of particles. This distribution has been called the ideal random mixture, one for which the location of any particle has no influence on the particle (or particles) that are adjacent to it. In other words, a particle that is removed from any location in the mixture has an equivalent chance of being of either species type. In practical terms, this distribution is often the best attainable for a real system of interacting particles.

one observes a distribution in the number of guests on each host, as well as free (unassociated) guests, leading to a less homogeneous outcome (Figure 15-2*b*).

15-2.1.3 Textured (Segregated) Mixtures. The most troublesome mixtures are those that exhibit long scale texture (i.e., segregation), complicating description of mixture distributions and characterization. Textured mixtures form when a characteristic of one or more particle species causes that component to separate into specific regions of the mixture, depending on the type of agitation applied to the bulk mixture. Also, dead zones or incomplete agitation of the powder can lead to segregated regions in blenders. In general, more free-flowing mixtures exhibit more extreme segregated states. Cohesivity acts to inhibit mixture segregation, as individual particles have trouble moving independently of the bulk mixture. Determining mixture quality of textured mixtures depends on accurately determining the size, location, and severity of the segregated regions.

Figure 15-3 shows two types of segregated mixtures, one for free-flowing materials and the other for a cohesive mixture. In the free-flowing case, if the particles do not differ in any particle characteristic other than color, long scale texturing of the mixture will not occur for a sufficiently agitated system. But when there are differences in particle characteristics (size, shape, density, etc.), a situation like that shown in Figure 15-3*a* can arise, where individual particle species are preferentially found in specific regions of the mixture (left to right in this case). For more cohesive systems, a partially randomized mixture can

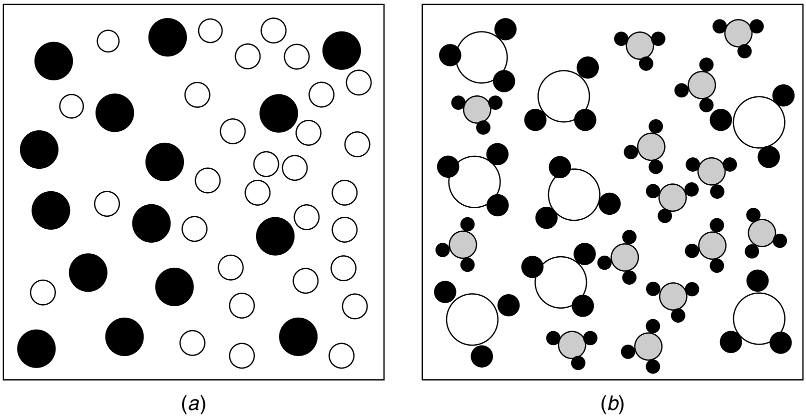


Figure 15-3 Distribution of particles differing in size for (*a*) freely-flowing and (*b*) cohesive materials.

develop, as shown in Figure 15-3*b*. This drawing depicts segregation of ordered units with different-sized carrier particles, but segregation of ordered units with leftover adherent particles is also possible.

In any real mixture there will be areas that correlate closely to many of the ideal distributions discussed previously. Unfortunately, the characterization of mixture quality cannot currently be done by viewing particle distributions throughout the mixture. For real systems, samples are extracted from specific regions of the mixture and it is important to ensure that the sample size is representative.

15-2.2 Powder Sampling

Real systems do not yield complete and pristine data on the distribution of particulate species within the bed. Instead, it is necessary to extract a finite, typically small number of samples from the mixture. These samples often have important limitations and biases, as discussed here. The most common means for sampling powder constructs is through the use of sampling thieves. These devices are inserted into the bed and extract samples from the interior. When devising a sampling scheme, it is important to adequately sample all regions of the powder bed. As mentioned, granular materials can segregate spontaneously, and can mix very slowly (especially when dispersion is the major mixing mechanism). Hence, sampling at only a few locations can lead to significant undersampling as regions of poor mixing are completely missed or underrepresented. Furthermore, postprocessing of a powder mixture can cause a previously well-mixed sample to de-mix and adversely affect further applications.

15-2.2.1 Physical Sampling Methods. The behaviors of two popular types of thief samplers are shown in Figures 15-4 and 15-5 (Muzzio et al., 1999). In Figure 15-4*a* we illustrate the bed disturbances that occur when using a *side-sampling thief*. This device consists of a tube with a slot in its side that can be

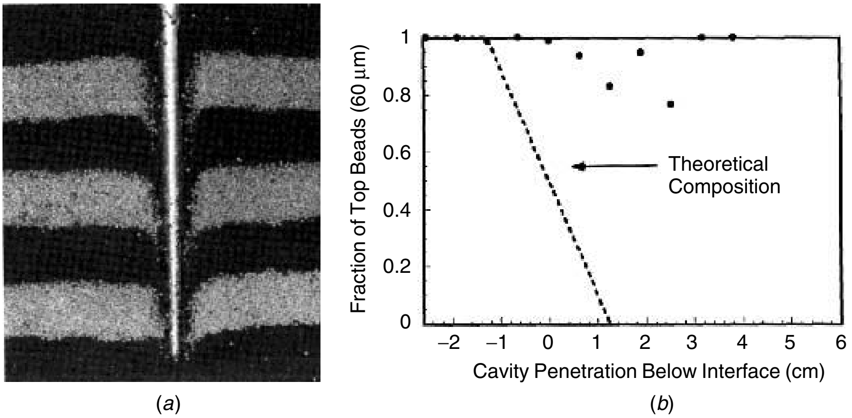


Figure 15-4 Systematic sampling errors introduced by a side-sampling thief. (*a*) Initially layered configuration of large (light) and small (dark) particles are noticeably disturbed as the thief entrains particles during insertion. (*b*) This type of thief relies on free flow of particles to fill a cavity when a slot is opened in the side of the sampling tube. Consequently, fine and freely flowing particles are overrepresented by this probe, and fine particles are transported to regions where they were not placed originally.

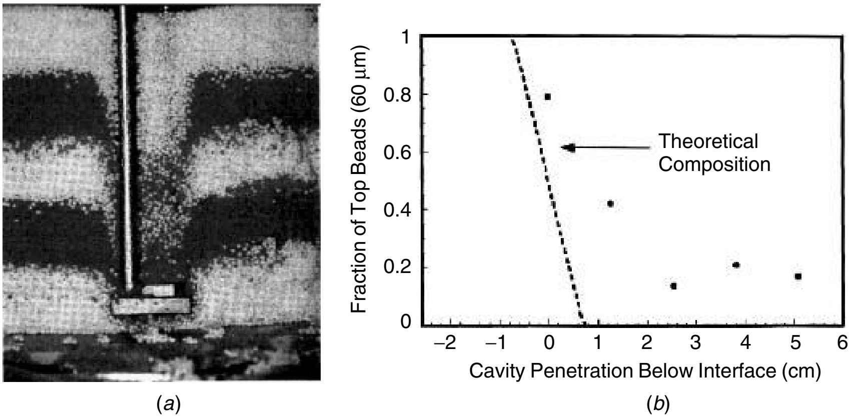


Figure 15-5 Sampling errors introduced by an end-sampling thief differ from those introduced by a side-sampling thief, but persist nonetheless. In this type of thief, a window is opened at the bottom of the sampling tube, and particles are forced into a cavity by further insertion of the thief. This eliminates the bias toward particles that passively fill a cavity more easily than others, but on the other hand, (*a*) these thieves entrain more particles during insertion, and (*b*) their performance again suffers from substantial systematic error.

opened to allow particles to flow into a cavity, and closed to extract the sample. An initially layered system of light gray 200 and dark 60 μm particles is visibly disturbed by inserting the probe. Particles are entrained along the insertion route, causing local particle rearrangements that typically result in the bed appearing to be anomalously well mixed. It is also significant that side-sampling thieves rely on particle flow into the sampling cavity to obtain particles; consequently, free-flowing or smaller particles can flow into the sampling cavity more readily than more cohesive or larger particles. These observations are quantified in Figure 15-4*b* that shows the fraction of smaller beads in samples obtained using a side-sampling thief in separate experiments in which 60 μm particles are initially arranged in a single thick layer over a bed of 200 μm particles. The thief obtains samples almost entirely consisting of the smaller species, irrespective of the actual concentration at the sampling location.

Sampling problems that arise from differences in particle flow into the sampling cavity can be mitigated through the use of *end-sampling thieves*, such as the one shown in Figure 15-5. For these thieves, the sampling tube is inserted to a desired depth in the bed, an aperture at the distal end of the probe is opened, and then the probe is pushed deeper into the bed to capture the sample; closing the aperture allows extraction of the sample. Particles are actively forced into the cavity rather than passively flowing into it, as in side-sampling thieves. Thus, this device is relatively free of differential sampling problems caused by differences in particle flowability. However, Figure 15-5*a* demonstrates that these devices are typically bulky and consequently entrain and disturb considerable material during their insertion. For the case discussed here, the resulting sample concentration measurements (Figure 15-5*b*) are improved over those of the side-sampling thief but remain very inaccurate, as data consistently overestimates mixture quality.

An alternative that is nearly free of either entrainment (Figure 15-4*a*) or flow (Figure 15-4*b*) anomalies is the *core sampler*. This sampler extracts an entire contiguous core of particles throughout the depth of insertion. At its simplest, the probe consists of a thin-walled tube that is inserted into a granular bed, together with a mechanized extrusion apparatus to permit samples to be extracted in a last-in, first-out manner after the tube has been removed from the bed. For capturing free-flowing particles, which can flow out of the tube, an end cap that can be opened during insertion and then closed during extraction is added to the device. Unlike the end-sampling thief, the end-cap mechanism here is internal to the sampling tube, and an entire core is extruded from the bed. The behavior of this device is demonstrated in Figure 15-6. Using the end cap (shown closed in Figure 15-6*a*), the concentration data obtained compare favorably with other methods, as shown in Figure 15-6*b*. Importantly, in the core sampler the core extends through the depth of the sampling tube, allowing for precise determination of concentrations between different layers of the bed. Furthermore, sample size is completely variable and can easily be adjusted for different mixtures, core sampler diameters, or changes in process parameters.

By foregoing use of the end cap, core sampler performance is improved further. In Figure 15-7 we display core sampling results for three different inner-diameter sampling tubes using a two-layer bed of common pharmaceutical excipient powders: microcrystalline cellulose and lactose. For all sampler diameters, the experimental data are indistinguishable from ideal expected concentrations. In practice, we note that it is important that the walls of the sampling tubes be

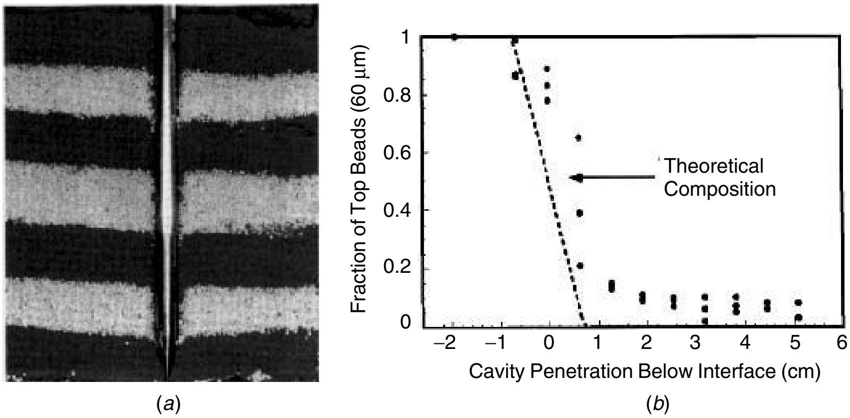


Figure 15-6 Core sampler with end cap can be used for freely-flowing (e.g., granulated) materials that would escape from the sampling tube during removal from the bed without the end cap. (*a*) Very little entrainment is visible after insertion, and (*b*) systematic errors are reduced. (From Robinson, 1999.)

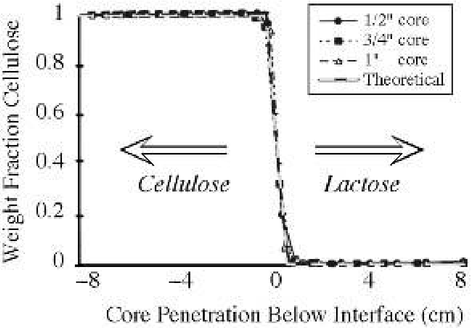


Figure 15-7 Non-free-flowing powdered material can be extracted from a blend using a core sampler with an open end. This case shows a sampling experiment using a thick layer of microcrystalline cellulose above a bed of lactose, where measurement errors are virtually undetectable.

polished (to prevent excessive entrainment and difficulty filling the tube during insertion) and that a well-regulated extrusion device be employed.

Once samples have been obtained, one can use a variety of available chemical, optical, spectroscopic, chromatographic, or other assays to determine concentration. For example, data in Figure 15-7 were obtained using a calibrated densitometric technique in which one of the two species was colored in advance. Similar results have been obtained using other assay techniques, such as reflection near infrared spectroscopy to evaluate concentrations of magnesium stearate (a common pharmaceutical lubricant) or conductivity assays to evaluate the mixing of salt (NaCl, KCl) in anionic excipients (Avicel).

15-2.2.2 Noninvasive Methods. Other, more technologically complex techniques have also been developed for visualizing the interior of granular beds. These include:

- *Diffusing wave spectroscopy*, where statistics of fluctuations in relatively thin, Hele–Shaw configurations are measured
- *Positron emission tomography*, where a single radioactive particle is tracked during flow within a granular bed using an array of external photomultipliers
- *Magnetic resonance imaging*, where magnetic moments of hydrogenated particles are aligned in structured configurations (e.g., stripes) and these structures are tracked for short periods of time
- *X-ray tomography*, where a population of radiopaque particles are tracked in a flow of interest

These techniques are typically expensive and cumbersome to implement; nevertheless, they reveal flows within an optically opaque bed and provide valuable information not available otherwise. For example, in Figure 15-8, we display results of x-ray tomography experiments that show the evolution of the interior mixing structure within a double-cone blender using molybdenum-doped tracer particles (dark in Figure 15-8). Data of this kind reveal a complexity in flow and mixing evolution that simultaneously represents the cause of historical difficulty in understanding the subject and the opportunity for future developments. As these methods are improved, they will yield more quantitative information about mixture quality, leading to more robust methods for characterization of powder mixtures.

15-2.3 Scale of Scrutiny

When extracting samples from sampling thieves, it is necessary to specify the sample size. This determination is based on the necessary scale of scrutiny for a particular system. Typically, this scale can be determined from the end use of

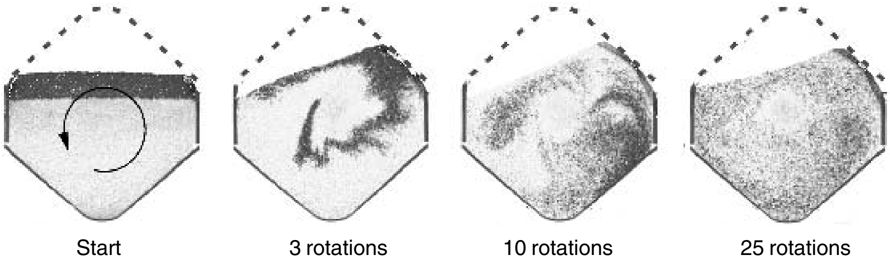


Figure 15-8 X-ray tomographic time series of blending of radiopaque grains in a double-cone blender is representative of several new techniques available for on-line and in situ assays of blending mechanisms. (From Chester et al., 1999.)

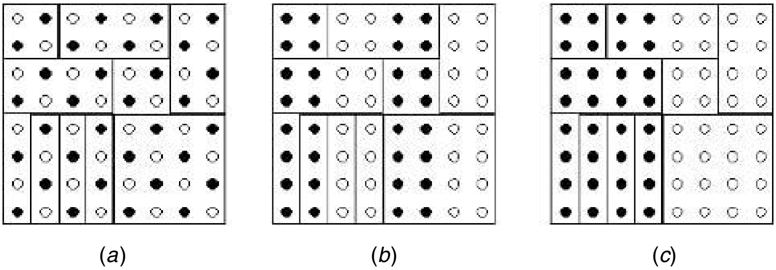

(a) (b) (c)

Figure 15-9 Three ideal mixture distributions are shown, representing (*a*) well-mixed, (*b*) striated, and (*c*) completely segregated mixtures. A number of boxes are drawn over each distribution to represent possible sample size and orientations. These posited sample locations show how sample size and location can adversely affect the true measure of mixture quality.

the product (e.g., pharmaceutical tablet). Component variations within a single dose generally do not affect the chemical uniformity of the final product units (mechanical properties are a different matter altogether). However, relying on too large a scale of scrutiny can mask mixing problems within a system.

Three model systems are shown in Figure 15-9. A series of boxes of varying size drawn over these particle distribution schematics illustrate some of the problems than can arise from improperly defining the scale of scrutiny. If the sample size is chosen as either the entire domain or a single particle, all the distributions shown in Figure 15-9 would have the same measure of mixing, which is clearly incorrect if the finished product has an intermediate scale. For other box sizes, we see that box size is largely irrelevant to the well-mixed case (Figure 15-9*a*), but the box size and orientation can have major influences on the apparent mixture distributions for the striated (Figure 15-9*b*) and completely segregated (Figure 15-9*c*) mixtures. For these mixtures, a symmetric 2 × 2 box encompassing four particles would appear to be the minimum scale of scrutiny necessary to extract reliable mixing information. The upper limit is bounded by a 4 × 4 box size, as the 4 × 4 box clearly would not detect the striated mixture in Figure 15-9*b*.

A key point to gather from the textured mixtures above is that the orientation of sampling can have a large impact on mixing measures as well as the sample size. Sampling perpendicular to a striated mixture must be undertaken with care to ensure that the sample size is smaller than the striation size to obtain accurate measures of mixture quality (or lack thereof).

15-2.4 Quantification of Solids Mixing: Statistical Methods

Clearly, the sampling protocol and extraction technique can have a major impact on the accuracy of the mixing measure. Once samples have been obtained, it then becomes necessary to develop mixing measures that give an accurate representation of mixture quality.

One of the most useful measures of mixedness is the intensity of segregation, I. This is essentially a normalized variance of concentration measurements. Intrinsic to the use of intensity of segregation therefore is the presumption that the mixing distribution is, at least to a first approximation, Gaussian. This raises two issues: (1) it is not clear that granular mixing tends toward a Gaussian state, and (2) in many practical applications a Gaussian is not the desired outcome. Indeed, in pharmaceutical processing, if a blend were Gaussian rather than uniform, the unavoidable presence of exponential tails on a Gaussian distribution would guarantee that some small fraction of tablets made from the blend would be beyond any therapeutic range that one could specify. Moreover, the expectation of a Gaussian distribution provides the manufacturer of a regulated product an incentive for extracting as few samples as possible, since a larger number of samples increases the probability of detecting product out of specification. Fortunately, granular flows appear to scatter particles more uniformly than a simple Gaussian would predict, although the details and mechanisms for this behavior are not yet well understood.

With these caveats in mind, the intensity of segregation, I, is defined as

$$\mathrm{I} = \frac{\sigma^2 - \sigma_\mathrm{r}^2}{\sigma_0^2 - \sigma_\mathrm{r}^2} \tag{15-1}$$

where σ^2 is the variance of sampled data, σ_r^2 is the variance of the same number of randomly chosen concentration data, and σ_0^2 is the variance of an initial, typically fully segregated state, again consisting of the same number of data points. Several forms of I appear in the literature; the form presented here is useful because it is normalized so that $\mathrm{I} = 1$ and $\mathrm{I} = 0$ correspond to completely segregated and randomly mixed states, respectively. In practice, values below 0.7 are rarely encountered, changing the range of expected values for good and poor mixed constructs.

Another mixing measure of importance to powder mixing is the relative standard deviation (RSD), also known as the coefficient of variance (CoV), defined to be

$$\mathrm{RSD} = \mathrm{CoV} = \frac{\sigma}{\mathrm{M}} \tag{15-2}$$

where σ is the standard deviation and M is the mean concentration over all samples taken. Often, in pharmaceutical applications, mixing protocols are written to ensure that a specified percentage of all extracted samples meet an RSD ceiling.

Related to the standard deviation of a mixture is the mixture variance. Variance measurements have the benefit of being additive, meaning that total variance can be subdivided into mixture variance plus sampling error, assay error, and so on. Using this quality, a more detailed analysis of bed variability can be obtained by separating the total variance measurement into separate dependent measurements. For example, for mixtures of cohesive and free-flowing components, it is very important to design stratified sampling schemes where multiple samples are taken from each of a series of predetermined sampling locations, allowing the experimenter to distinguish within-location and between-location variability. For tumbling blenders it is extremely useful to divide the measured variance into axial variance and radial variance components. Axial variance measures the differences in concentrations between sampling locations, while radial variance measures variance within the bed at a single location. Using a core sampler greatly aids these measurements, as concentration data from a single core and average values between different cores can be used separately. Formally, for each core j,

$$\overline{x_j} = \frac{\sum_i x_{ij}}{N_i} \tag{15-3}$$

where the core mean concentration is $\overline{x_j}$, x_{ij} is a given sample concentration, and N_i is the number of samples in that core. The standard definition of variance is

$$\sigma^2 = \sum_j \sum_i \frac{(x_{ij} - \overline{x})^2}{N} \tag{15-4}$$

where σ^2 is variance, N is the number of samples, and $\overline{x}$ is the mean composition. Substituting eq. (15-1) into eq. (15-2) and rearranging leads to

$$\sigma^2 = \frac{1}{N} \sum_j N_i(\overline{x_j} - \overline{x})^2 + \frac{1}{N} \sum_j \sum_i (x_{ij} - \overline{x_j})^2 \tag{15-5}$$

In eq. (15-5) the first term is a measure of axial variance (σ_A^2) and the second term, radial variance (σ_R^2). These two measures give a more accurate description of mixing quality within a granular blend than can be achieved with any single measurement.

15-3 THEORETICAL TREATMENT OF GRANULAR MIXING

Research into granular flow and mixing can be roughly divided by chronology: prior to 1990, industrially usable results were mostly empirical (e.g., in experiments using a particular blend in a specific device), and fundamental research was largely analytic (e.g., using continuum approximations to the granular state applicable only to one phase of granular behavior). Although significant progress has been made into developing specialized engineering solutions as well as models of fundamental behaviors of ideal granular systems, little generally applicable knowledge was attainable using either approach. Over the past decade, computational and methodological advances have permitted quantitative evaluations of granular flow, transport, and mixing at a much greater level of detail. In this section we review progress on tumbling flow and blending phenomenology that has led to the development of the best existing predictive models.

15-3.1 Definition of the Granular State

A chief limitation, and the principal area of opportunity for the future, in developing predictive understanding of granular flows is the coexistence of multiple, history-dependent granular states. Within a device—be it a tumbler, a mill, a fluidized bed, or a high-shear intensifier—granular material can, and typically will, exhibit multiple rheologically different phases that vary nontrivially and often with profound consequences as a function of minor changes in material or operational parameters. This is a particular problem in the pharmaceutical industry, where products may be developed in dry northern latitudes, and produced in wet equatorial climates. Both hygroscopic excipients and actives behave very differently in these two environments, and blending regimens that work in one may well fail in the other. Moreover, even within a single well-controlled bench scale device, multiple phases are typically present. The tumbling blender is a case in point.

In Figure 15-10 we display a deceptively common outcome of an attempt to blend dissimilar materials, here grains differing only in size and color. In this transparent 4 L capacity[1] V-blender, we have tumbled equal volumes of smaller light-gray and larger black grains at 6 rpm for 200 revolutions. The visibly segregated state is one of several distinct segregated configurations that

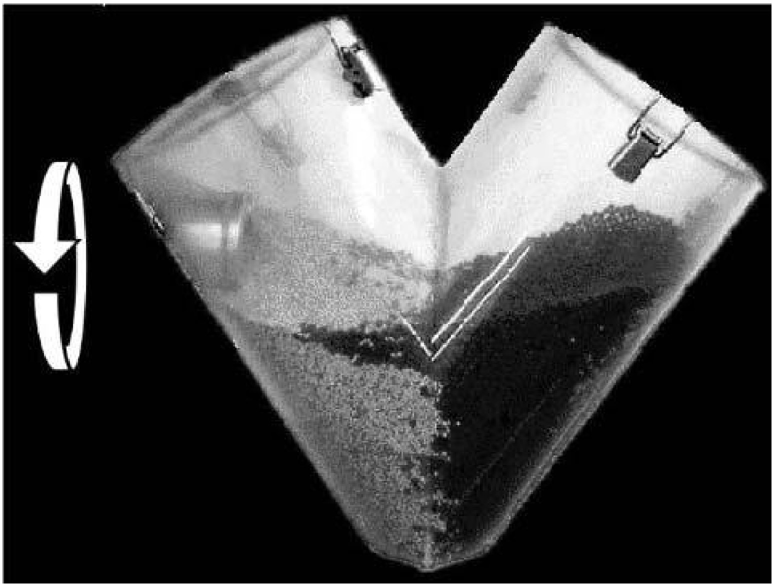


Figure 15-10 Left–right segregated state, here in a transparent V-blender, between larger (dark) and smaller (light) grains. This state occurs spontaneously at high fill levels and fast tumbling speeds in many tumbler designs.

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[1] The reader should note that capacity customarily refers to a fraction (generally, 60%) of the total interior volume of a blender.

form spontaneously and reproducibly in all common blender geometries and scales. Once formed, these patterns persist despite the practitioner's best efforts at modification of process parameters. Developing cures for this type of problem demands a systematic understanding of why de-mixing occurs in the first place, so that the cause of segregation can be addressed directly. This understanding, in turn, requires an analysis of the different granular behaviors seen during the tumbling operation.

A first step in the analysis of granular behaviors is the characterization of the different granular phases that are inevitably present during flow. It has been recognized for over a century that grains, unlike common fluids, must dilate in order to flow—that is, grains in the static state are interlocked and cannot move without separating (see Figure 15-11*a*). The locations and timing of flow can be quite complex; nevertheless, far from flowing regions a granular bed remains static and solidlike, whereas near a shear interface, grains are fluidlike or gaslike as the shear rate increases. The modifier "like" is important to include, for a solidlike region is not truly elastic, as it transmits stress along irregular compressive chains, it undergoes slow creep and settling on time scales ranging from seconds to hours, and it can solidify into a rigid cake over time scales of days to months. Similarly, the fluidlike phase transmits shear discontinuously in both space and time and does not obey Navier–Stokes equations, and the gaslike phase is far from equilibrium and is not characterized by Maxwell–Boltzmann statistics. The selection of these three latter phases—glassy, fluidlike, or gaslike—in a specified location depends on details of the bed dynamics, including the rate of shear, the extent of compaction of the bed, and the geometry in which the bed is confined. It is the differences both between qualitative behaviors of different regions of a granular bed at different times and between any one of the behaviors and accepted models for flow and dispersion that make predictive understanding of even the simplest granular systems challenging.

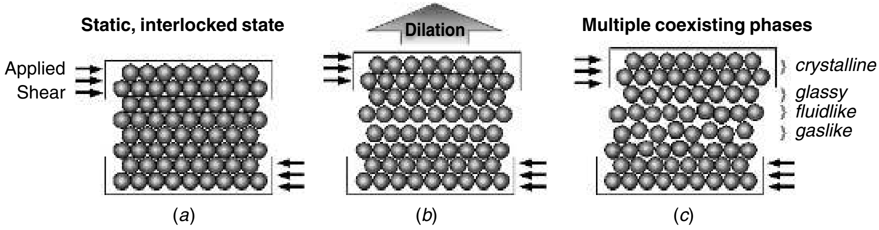


Figure 15-11 Schematic of dilation mechanism that is a prerequisite for the flow of solids. (*a*) In an undisturbed state, grains are interlocked and behave much like an ordinary solid. (*b*) A granular bed dilates in response to applied shear and can then flow. (*c*) In the flowing state, the bed can form distinct crystalline, glassy, fluidlike, and gaslike phases. The crystalline phase is regular and ordered, the glassy phase is disordered but static, the fluidlike state flows but exhibits enduring contacts, and the gaslike state is characterized by rapid and brief interparticle contacts.

The ability of granular systems to coexist in rheologically distinct states has many consequences. It has prevented the scientific community from developing effective devices for measuring rheometric properties. Essentially all available techniques provide an average measure of strongly nonlinear, strongly variable behavior. Although accumulated experience can help the practitioner use this type of information within a narrow range of parameter variability, in practice a lack of rheometric techniques is perhaps the main reason for a lack of effective constitutive models. Models abound, but their experimental verification is somewhere between unfeasible and impossible. As a direct consequence, computer simulation techniques for granular flows are much less developed than those used for fluids, and our ability to develop granular flow systems *in silica* is largely a goal for future generations.

Nonetheless, despite intrinsic difficulties in developing all-encompassing models for granular flow, important blending problems of practical interest have been effectively solved using analytic, computational, and semiempirical means. We summarize the current understanding of granular blending and de-mixing in subsequent sections.

15-3.2 Mechanisms of Mixing: Freely-Flowing Materials

In tumbling applications, dilation and flow principally play out near the unconstrained upper surface of a granular bed, and except for solid-body rotation, the bulk of grains beneath are thought to remain nearly motionless during rotation of the blender. This simplified picture changes for some blenders (notably the V-blender, in which flow is strongly intermittent; see Moakher et al., 2000), but predictive models for blending in most common blending geometries can be derived by disregarding all transport beneath the free surface. In the sections following, we summarize the best existing models and methods and describe their application to common tumbler designs. A useful design choice for the purposes of illustration is the horizontal drum tumbler. The horizontal drum is used in many chemical, metallurgical, and pharmaceutical industries in the form of ball mills, dryers, rotary kilns, coating pans, and mixers. Flow in rotating drums with increasing tumbling speed has been described qualitatively in terms of regimes termed: slipping (or slumping), avalanching, rolling, cascading, cataracting, and centrifuging. These are defined as follows.

15-3.2.1 Slipping. The slipping regime occurs when the granular bed undergoes solid body rotation and then slides, usually intermittently, against the rotating tumbler walls. This occurs most frequently in simple drums that are only partially filled and is typically counteracted by including baffles of various designs along the inner walls of the tumbler. While the slipping regime is not important for blending purposes per se, it is encountered even in effective blending systems, and an evaluation of the number of times a bed turns over per tumbler revolution will often reveal the presence of some slipping.

15-3.2.2 Avalanching. A second regime seen at slow tumbling speeds is avalanching flow, also referred to as *slumping*. In this regime, flow consists of discrete avalanches that occur as a grouping of grains travel down the free surface and come to rest before a new grouping is released from above. The avalanching regime is not seen in tumblers larger than a few tens of centimeters in diameter, but it is an instructive case because a flow and mixing model can be derived in closed form for simplified drum geometries.

To analyze this problem, one needs only observe that if the angle of repose at the free surface immediately before an avalanche is θ_i, and after an avalanche is θ_f, the effect of the avalanche is to carry a wedge of material in the angle $\theta_f - \theta_i$, downhill, as sketched in Figure 15-12*a* for an idealized two dimensional disk blender. The same behavior occurs for all fill levels, and one can readily use this model to make several concrete predictions. First, mixing occurs during avalanches through two distinct mechanisms: (1) particles within a wedge rearrange during a single avalanche, and (2) particles rearrange globally between wedges during successive avalanches. Second, at 50% fill (Figure 15-12*b*) no two avalanching wedges intersect, so no global mixing between separated regions can exist, and mixing must slow. Third, since flow occurs only near the avalanching surface, at high fill levels a nonmixing core necessarily develops (Figure 15-12*c*). Although this model is oversimplified and neglects material variations, boundary effects, and other important phenomena, these conclusions carry over to more realistic tumbling systems.

15-3.2.3 Rolling. At higher tumbling speeds, discrete avalanches give way to continuous flow at the surface of a blend (Shinbrot et al., 1999a). Grains

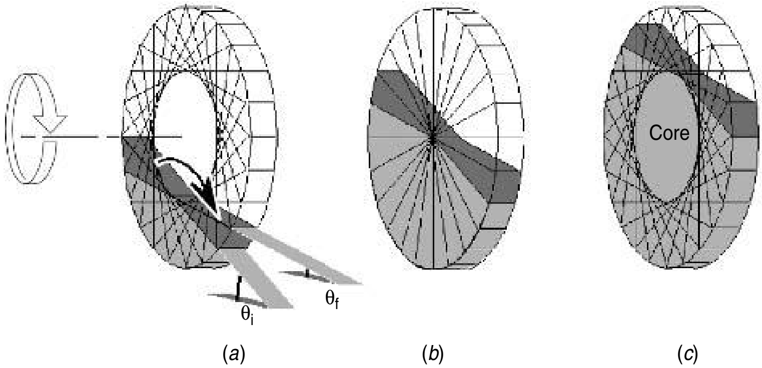


Figure 15-12 (*a*) Avalanching flow in an idealized disk tumbler transports grains from an uphill wedge to a downhill wedge as the free surface relaxes from an initial angle, θ_i, to a final angle, θ_f. This implies that global mixing occurs in quadrilateral regions where grains within one wedge intersect with a second wedge. (*b*) Consequently, tumblers mix more efficiently at low fill levels than at high, and global mixing nearly stops at 50% fill. (*c*) At fill levels above 50%, a core develops that does not visit the avalanching surface and therefore does not experience transport or mixing.

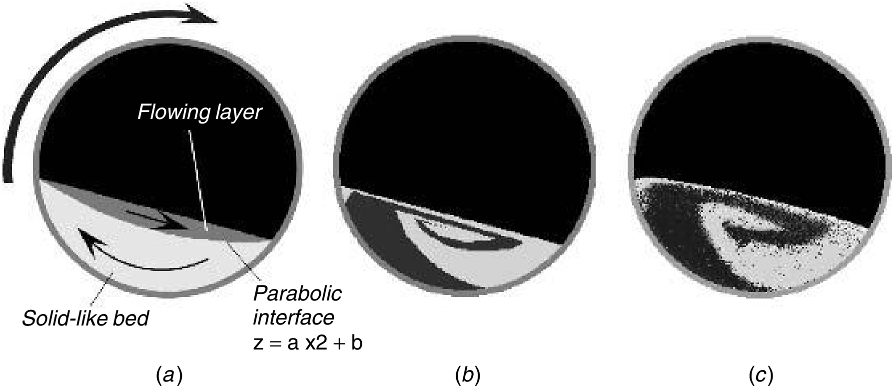


Figure 15-13 (*a*) In the rolling regime, the blend separates into a flowing layer near the surface and a solidlike supporting bed. (*b*) By establishing simple conditions such as mass conservation, one can generate an analytic model for the flow, producing mixing patterns between initially separated and different-colored but otherwise identical grains. (*c*) Comparison with experimental mixing patterns using freely flowing grains in a small drum tumbler reveals substantial agreement. The snapshot in part (*c*) is obtained from the interior of the blend using a solidification technique.

beneath this surface flowing layer rotate nearly as a solid body with the blender until they reach the surface. One can solve for flow and transport subject to certain simplifying assumptions in this regime as well. For this solution, one assumes that the grains are so small as to be regarded as a continuum and one takes the free surface to be nearly flat, as sketched in Figure 15-13*a*. The interface between the flowing layer and the bed beneath has been determined experimentally and computationally to be roughly parabolic in shape, and by demanding mass conservation at this interface, one can construct continuum flow equations for this system. If one simulates the mixing in an idealized disk blender of mechanically identical grains initially separated by color to left and right of a vertical central plane, one obtains the results displayed in Figure 15-13*b* (for a particular fill level and flowing layer depth). Corresponding experimental results are shown in Figure 15-13*c*.

15-3.2.4 Cascading, Cataracting, and Centrifuging. For larger tumblers, or for tumblers rotated at higher speeds, the surface is manifestly not flat, as shown in Figure 15-14 in a 1 m diameter disk tumbler. This flow, termed *cascading*, differs qualitatively from the rolling flow solution; here the flowing layer is thin, is nearly uniform in speed and thickness, and has been modeled as depth-averaged pluglike flow. As the rotation speed of the tumbler is increased, the surface becomes increasingly sigmoidal until grains become airborne, and at higher speeds yet, the grains centrifuge against the tumbler wall. These regimes are termed *cataracting* and *centrifuging*, respectively, and have not been well analyzed.

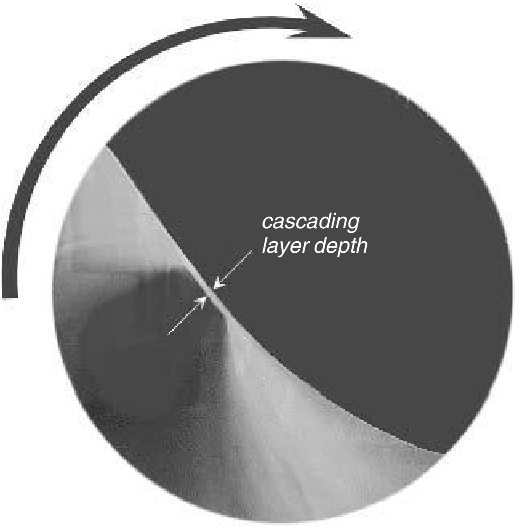


Figure 15-14 Cascading flow occurs in large tumblers or during tumbling of fine but freely flowing grains. This snapshot shows a 1 m diameter 1 cm wide transparent disk tumbler partially filled with colored ∼500 μm irregular grains. Initially, light and dark grains were placed to the left and right of a central plane; this snapshot shows the mixing pattern at one-half revolution of the disk. This tumbler is thin, so grains are under the strong influence of wall effects; nevertheless, this example serves to illustrate that the free surface is manifestly not flat, and the cascading layer is thin and nearly uniform along the flowing surface.

15-3.3 Mechanisms of Mixing: Weakly Cohesive Material

Another mechanism of granular and powder mixing is associated with blending of weakly cohesive materials. Weakly cohesive materials (e.g., powders and fine grains in the size range 50 to 300 μm) exhibit stick-slip motion so that flow becomes intermittent rather than continuous. This is a situation of practical importance since most industrial applications use particles across a broad range of sizes and materials. As the size of grains diminishes or as interparticle cohesion grows, stick-slip flow transforms mixing interfaces from a smooth, regular patterns as shown in Figure 15-15 (500 or 700 μm cases) to a complex, irregular pattern, shown in Figure 15-15 (300 or 100 μm cases). In simple geometries this response to shear can be modeled accurately: If we assume that the flowing surface of a bed sticks and slips periodically, the mechanism displayed in Figure 15-13*a* can be embellished by allowing the shear band between flowing layer and bed to deform periodically (Brone et al., 1997). This produces mixing patterns between initially separated but identical grains that are substantially similar to experimentally observed ones, as shown at the bottom of Figure 15-15. This is important for blending because in smooth regular flow, adjacent particles remain nearby for long periods of time, while

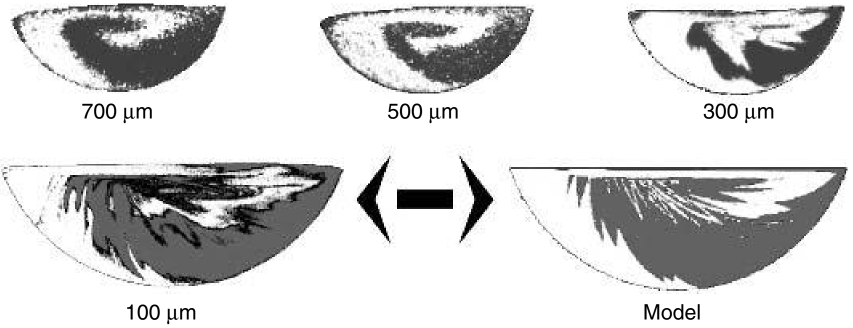


Figure 15-15 Mixing patterns after one revolution in identical drum tumblers loaded with identical (except for color) grains in four experiments using successively finer grains as well as in a model simulation of idealized stick-slip flow. At 700 and 500 μm, the mixing interface remains smooth and regular; below about 300 μm, it becomes variegated due to intermittent slipping of the cascade. Each experimental snapshot shows a view from the interior of a blend using the solidification technique described by Wightman et al. (1995), and all cases began with light grains to the left of center and dark grains to the right.

in intermittent stick-slip flow, particles can rapidly relocate across the blender, resulting in an exponentially rapid growth of interfaces between separated regions of grains (Shinbrot et al., 1999b).

For particles smaller than about 100 μm, cohesive forces (believed to be due to van der Waals interactions for intimate contacts, and to surface tension of adsorbed water layers for lubricated contacts) between particles become comparable to particle weights, and small particles can stick to one another in relatively rigid aggregates. Unless such aggregates are destroyed, the system will behave as if it had an effective particle size much larger than the primary particle size.

For strongly cohesive materials, it is typically necessary to fragment agglomerates through the introduction of high-shear, intensification devices, such as impellers or mills that energetically deform grains on the finest scale. Many forms of intensification are used in industrial practice. Some common approaches include passing the blend through shaker sieves or through hammer or pin mills between blending stages, as well as using high-speed devices within blenders, such as intensifier bars in tumbling or choppers in high-shear granulator-style mixers.

Essentially no detailed systematic quantitative information is available concerning the effects of intensification on blend quality. We are aware of no studies investigating the micromixing quality as a function of intensity and duration of applied shear. Scale-up and design information provided by equipment vendors is largely limited to advising the user to keep intensifier tip speed and time of operation constant during scale-up. Although this guideline is reasonable in lieu of rigorous information, it is clear that in situ intensifiers apply shear only locally, and nonuniformly, to the mixture; the end result is almost guaranteed to be affected by the interplay of the intensity of the shear field, the residence time of particles in the shear field, and the global homogenization capabilities of the blender. At the present time, laboratory devices for applying shear uniformly and at a known rate are unavailable, making study of the problem even harder. Given a tendency across industry to deal with ever smaller, ever more cohesive materials, understanding the role of shear on blend quality is undoubtedly one of the areas in greatest need of attention by the scientific community.

15-3.4 De-mixing

Processing blends of dissimilar grains almost invariably promotes *de-mixing*, also referred to as *segregation*, characterized by the spontaneous emergence of regions of nonuniform composition. Segregation due to differences in particle size in a blend has drawn the greatest attention in the literature, including studies of fluidized beds, chutes, hoppers, vibrated beds, and tumbling blenders, but segregation due to differences in particle density, shape, and triboelectric order have also been recorded. As a practical matter, segregation manifests itself in granular mixing that characteristically improves over a brief initial period, while convection generates large scale mixing, and then degrades, often dramatically as slower segregational fluxes take over. De-mixing should not be confused with the phenomenon of overblending, which is also frequently encountered in blending applications. Overblending is associated with physical degradation of material properties, as occurs, for example, when a waxy lubricant is excessively deformed, causing it to coat pharmaceutical grains and reduce their bioavailability, or when coated granules are damaged through abrasion or fracture.

At the present time, mechanisms for segregation, even in the simple tumbling drum, remain obscure, and work on more complex and industrially common blender geometries is extremely limited. Three distinct types of de-mixing are moderately well characterized in tumblers: radial de-mixing, axial de-mixing, and competitive patterned de-mixing. We describe each of these in turn.

15-3.4.1 Radial De-mixing. Segregation typically proceeds in two stages. First, large grains rapidly segregate radially, producing a central core of fine grains surrounded by larger grains, identified in Figure 15-16 for a simple drum tumbler. Unlike the core seen in overfilled tumblers, this core appears at fill levels under 50% and is associated exclusively with migration of fine grains toward the center of an overturning blend. Radial segregation is seen in both quasi-2D and fully 3D blenders of various geometries. In simpler 3D geometries, such as the drum, double-cone, or tote, the core is nearly always apparent when blending significantly dissimilar grains, while in more complicated geometries such as the V-blender or slant cone, the core becomes significantly distorted and may only be conspicuous for higher fill levels or in certain (e.g., upright) orientations of the blender. Even in the simplest case of the drum tumbler, however, the location and dynamics of the core remain somewhat enigmatic—for example, as shown

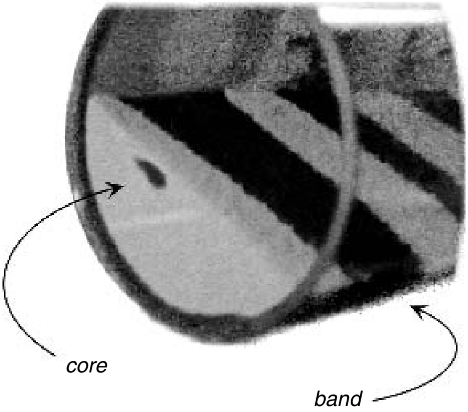


Figure 15-16 Typical segregation pattern seen between fine (dark) and coarse (light) grains in a small transparent drum tumbler. A core of fines travels along the entire length of the tumbler, connecting the bands that emerge at the surface in a single bulging tube. The coarse grains are constrained to flow within the confines defined by this tube. This constraint is important for understanding mechanisms of de-mixing in more complex geometries, as summarized in Section 15-3.4.

in Figure 15-16, the core is actually located upstream of the geometric center of the granular cascade.

The core appears to form as a result of two cooperative influences. First, smaller grains percolate through the flowing layer to occupy successively lower strata each time the bed overturns. Second, once a sufficient volume of smaller grains has accumulated, the larger grains tend to roll increasingly freely over the (comparatively smooth) substrate of smaller grains. This higher-speed surface flow reinforces the segregated state by expelling remaining slower small grains. These mechanisms are very robust, and cores are almost invariably found in tumbling of freely flowing grains with diameter ratios between about 1 : 1.5 and 1 : 7. As the diameter ratio approaches unity, the core becomes more diffuse, while as the diameter ratio grows sufficiently large, fine grains can percolate increasingly freely through a matrix of larger grains or, if sufficiently fine, can coat the larger species.

15-3.4.2 Axial De-mixing

A second stage of segregation occurs in drum tumblers as grains in the core migrate *along* the tumbling axis. Numerical and experimental investigations have attributed this migration to conflicting causes (e.g., a secondary flow within the core leading to a bulging of the core toward the surface versus different angles of repose of fine, mixed, and coarse grains). Whatever the ultimate cause, the result of this axial migration is the formation of a series of bands as shown in Figure 15-16. In this final state, two pure phases of material are formed, divided by sharp boundaries with very little intermixing.

15-3.4.3 Competitive Patterned De-mixing. In more complex, and more common tumbler geometries, several distinct segregation patterns have been observed. These patterns are believed to arise from a competition between surface segregation of coarse grains flowing over a radially segregated core of fine grains and interactions with the boundaries of the tumbler. Despite significant differences between common blender geometries, there is substantial commonality in the ultimate patterns seen. For example, mixing of large, light-gray, and small, dark-gray grains in a double cone and a V-blender generate similar patterns in both experiments and particle-dynamic simulations (described in Section 15-4), as shown in Figure 15-17.

As parameters such as fill level, tumbler speed, and concentrations of the different particle species are varied, the patterns observed change significantly. Importantly, there appear to be few dominant and recurring patterns that are seen in both experiments and simulations in all blender geometries. Notably at high fill levels and tumbling speeds, the left–right state shown in Figure 15-10 appears to dominate. This pattern and two other common variants are shown at the top of Figure 15-18 in top views of the surface of a double-cone blender. Each of these patterns appears reproducibly and spontaneously whenever different-sized grains are tumbled in any of several blender geometries. Simulations shown beneath the experimental figures in Figure 15-18 use a continuum model in which large

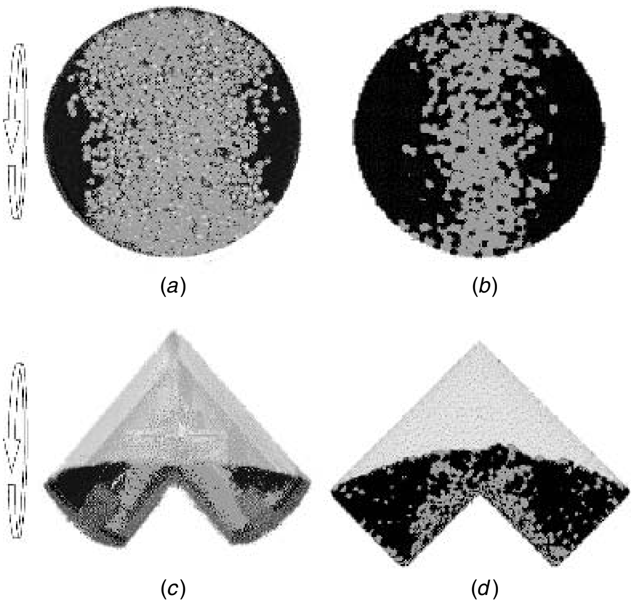


Figure 15-17 Axial segregation in top views of double-cone blender from (*a*) experiment and (*b*) particle-dynamic simulation using large, light and small, dark spherical grains. Similar patterns are seen in other tumbler designs: for example, in the V-blender in (*c*) experiment and (*d*) simulation.

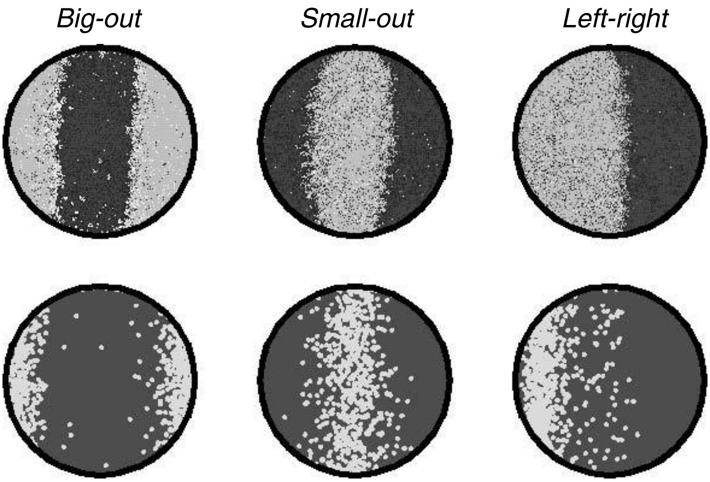


Figure 15-18 Three common segregation patterns between large (light) and small (dark) grains seen in top views of a double-cone blender. Top: experimental snapshots; bottom: simplified continuum simulations.

particles are convected on the surface of an idealized convex bed of smaller grains. Container geometry is included by assuming that large particles rebound specularly when they reach the downstream boundary of the idealized blender. Correspondence between experimental data and this simulation indicates that ongoing improvements in modeling show promise for unveiling the underlying mechanisms of de-mixing and permitting eventual accurate modeling of practical granular processing systems.

15-4 BATCH MIXERS AND MECHANISMS

15-4.1 Tumbling Mixers

Although drum blenders represent a convenient paradigm for the purpose of categorizing granular behaviors, most blending operations occur in more complex tumbler geometries. Three of the most common geometries used in pharmaceutical operations are the double cone, the V-blender, and the bin blender, sketched in Figure 15-19. Each of these geometries possesses many variants; for example, symmetry can be broken to introduce cross-flow by slanting the double cone, by elongating one of the arms of the V-blender, or by inserting baffles in a bin.

To model flow and blending in complicated geometries, particle-dynamic simulations have been applied. In these simulations, particles are treated as individual entities with physical properties (e.g., size, static and dynamic friction coefficients, coefficient of restitution, etc.) appropriate to the problem of interest, and Newton's laws of motion are integrated for each particle. Particle-dynamic simulations are similar in concept to molecular-dynamic simulations but include

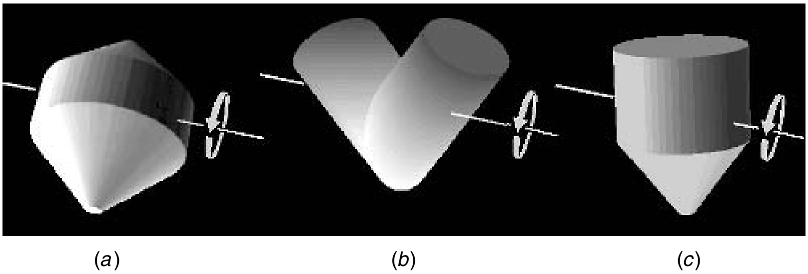


Figure 15-19 Three common tumbler designs: (*a*) double cone, (*b*) V, and (*c*) tote or bin blenders. Video clips of these mixers are provided on the Visual Mixing CD affixed to the back cover of the book.

features of importance to the flow of macroscopic particles (e.g., static and dynamic friction models) in place of microscopic properties (e.g., bond strengths and chemical potentials). Particle-dynamic simulations come in many different types, depending on how they treat physical parameters, such as rolling friction and particle shape, or numerical issues, such as search algorithms and routines to maintain computational stability. As such, results of distinct computational simulations can differ, sometimes significantly, and the importance of experimental validation of numerical results cannot be overemphasized.

Two of the most common classes of particle-dynamic simulations are termed hard-particle and soft-particle methods. *Hard-particle methods* calculate particle trajectories in response to instantaneous, binary collisions between particles, and allow particles to follow ballistic trajectories between collisions. This class of simulation permits only instantaneous contacts and is consequently often used in rapid flow situations such as are found in chutes, fluidized beds, and energetically agitated systems. *Soft-particle methods*, on the other hand, allow each particle to deform elastoplastically and compute responses using standard models from elasticity and tribology theory. This approach permits enduring particle contacts and is therefore the method of choice for tumbler applications. The simulations described in this chapter use soft-particle methods and have been validated and found to agree in detail with experiments.

15-4.1.1 V-Blender. Mixing in all tumbling blenders consists of a fast convective stage, driven by the mean velocity of many particles, followed by a much slower dispersive stage, caused by velocity fluctuations leading to rearrangements of individual particles. Convection in grains (as in fluids) is by far the faster and more efficient mixing mechanism, yet at the same time it suffers from the same mixing limitations known for fluids: convective flows can—and very often do—possess barriers to mixing (e.g., islands) that do not interact with surrounding material. Two pathologies are readily observed: overfilled mixers develop elliptic, nonchaotic islands that rotate as a unit in the center of the granular bed (discussed in Section 15-3.2.2), and symmetric blenders (seen in most standard designs) exhibit separatrices that divide the flow into noninteracting

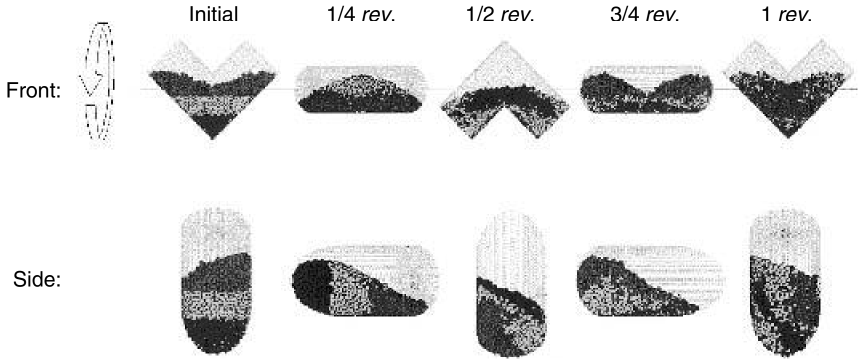


Figure 15-20 Rapid convective flow seen in particle-dynamic simulation of identical but colored spheres in a V-blender. Top: Front view reveals that unlike in some designs, convection in this blender drives grains axially, alternately outward toward the tumbler arms and inward toward its center. This axial flow strongly influences mixing. Bottom: Side view indicates that transport is dominated by a spiraling flow, seen also in drums and other blenders (cf. Figure 15-13). The full simulation is included on the Visual Mixing CD affixed to the back cover of the book.

sectors. Beyond this, little is currently known of details of particle flow patterns and mixing barriers in practical, three dimensional blender geometries, although there is strong evidence indicating that flow bifurcations analogous to those seen in fluids may be present in granular tumblers.

Convection in the context of granular blenders refers to transport associated with flow driven by gravity (in tumbling blenders) or impellers (in intensified, ribbon, or other blenders). Convection is observed in all functioning blender geometries and can be visualized using particle-dynamic simulations. In Figure 15-20 we display successive front and side views taken a quarter revolution apart of 20 000 identical but colored spheres tumbled in a V-blender in the cascading regime. These snapshots illustrate the qualitative motion produced in this blender, which causes the bed to overturn from top to bottom. Mixing due to convective flow grows linearly with time insofar as the area of an interface (e.g., between differently colored layers in these snapshots or in Figure 15-13*b* and *c*) grows characteristically linearly with time. Similar qualitative behaviors are seen in all tumbler geometries, although the quantitative mixing seen can differ considerably between geometries (Moakher et al., 2000).

15-4.1.2 Bin Blender.

In contrast to convection, which can effectively intersperse grains in a tumbler within tens to hundreds of revolutions, is *dispersion*, or *diffusion*. Dispersion refers to the random relocation of individual grains due to collisions between adjacent particles and can take hundreds to thousands of revolutions to act. Thus, particles can only cross a plane separating the two arms of the V-blender (or an equivalent symmetry plane in many other blender geometries) as a result of occasional collisional happenstances and not

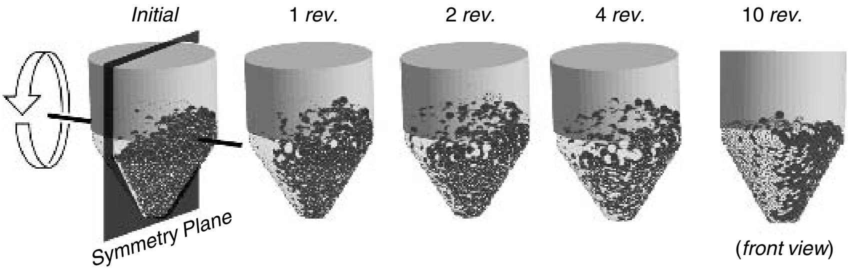


Figure 15-21 Dispersive mixing is slow across the symmetry plane of a blender, here a bin design. After 10 revolutions, a front view reveals clear evidence of the initial left–right distribution of identical but colored spheres in this particle-dynamic simulation. The full simulation is included on the Visual Mixing CD affixed to the back cover of the book.

as a result of an overall mean flow. Various stratagems, including the use of baffles, asymmetric cross-flow designs (referred to earlier), irregular rotation protocols, and axial rocking, have been introduced to mitigate this limitation. Notwithstanding these improvements, dispersion is the rate-limiting mechanism for mixing, and there is much potential for improvement of dispersive mixing.

Although convection is typically orders of magnitude more rapid than dispersion, the relative contribution of each mechanism to blending is strongly influenced by the initial distribution of species in the mixer. Thus, ingredients loaded in horizontal layers (as in Figure 15-20) can be mixed relatively rapidly, while ingredients layered side by side, either intentionally (as in Figure 15-21) or inadvertently (as a result of careless loading of a tumbler), will typically mix enormously more slowly.

To visualize this effect, in Figure 15-21 we display dispersive mixing of 8000 identical but colored grains loaded side by side, in a bin blender. With each successive revolution, only a few particles cross the interface separating the two symmetric halves of the tumbler, and as a result, after 10 revolutions the original particle ordering is still unmistakable. Systematic assays obtained from experiments of blending of realistic pharmaceutical excipients and actives confirm that imperfectly loaded blends retain any initial asymmetry for many hundreds of tumbler revolutions.

15-4.2 Convective Mixers

The second class of blenders commonly used in industrial applications is convective blenders. In contrast to tumbling blenders, convective blenders primarily mix by transporting material throughout a mixing vessel by the motion of a stirring device. A typical convective blender consists of a stationary chamber swept out by stirring mechanisms, such as rotating impellers. Convective blenders have a broad range of applications and can be used to blend components that cannot be adequately combined in tumbling blenders, such as materials that are prone to segregate or agglomerate. Many convective blenders can be designed to accommodate continuous rather than batch processing, further adding to their utility.

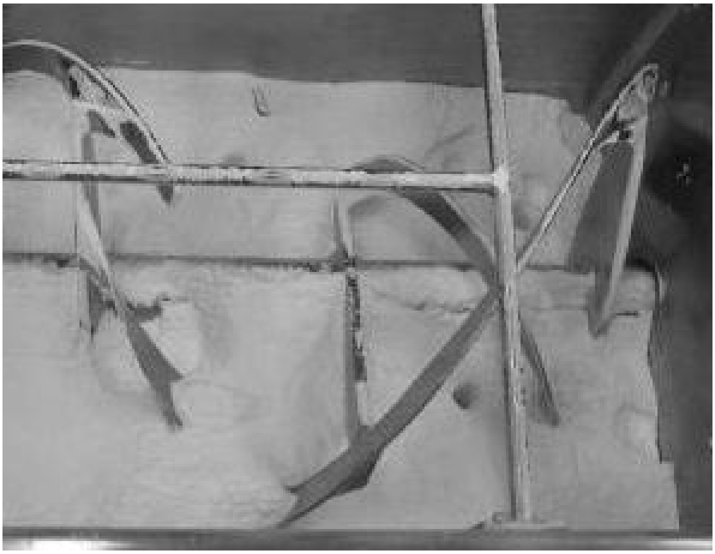


Figure 15-22 Well-mixed powder bed in a five-spoke ribbon blender, with both sampled cores and regions blocked by mixing blades visible.

However, despite this versatility, we have only a very limited understanding of the dynamics and performance of convective blenders. This is due to both the inherent difficulty involved in characterizing powder and granular mixing and the fact that few researchers have investigated these devices (see, e.g., Masiuk, 1987). In fact, much of the limited quantitative analysis was performed over 30 years ago (Adams and Baker, 1956; Greathead and Simmons, 1957; Poole et al., 1964; Ashton and Valentin, 1966; Harnby, 1967; Williams and Khan, 1973) and suffers from many of the limitations and difficulties of powder bed and granular mixing characterization discussed elsewhere in this chapter. Convective blenders also pose special challenges to powder bed analysis, as the impellers may hinder or block acquisition of powder samples, rendering thorough and uniform sampling difficult. Figure 15-22 shows a blended powder bed in a ribbon blender, a common convective blender. Several cores have been extracted from this bed, but it is evident that certain locations were impossible to sample, due to blades obstructing that location. Process design and optimization utilizing convective blenders are therefore performed predominately on a case-by-case basis.

15-4.2.1 Blending Mechanisms. Powder blending in a convective mixer is accomplished primarily by convection and shear effects. The motion of the impeller initiates blending by transporting material from region to region within the mixing chamber. This motion also generates slip planes, which often results in nonnegligible shear mixing as well. Diffusive mixing typically has a minimal role in mixing in these blenders. This combination of mixing mechanisms produces superior results in many applications. Segregation of particles of different properties can be a major problem in tumbling mixers, potentially resulting in de-mixing or agglomeration (accretion of smaller particles into larger clumps). The motion of a convective mixing impeller both prevents the formation of and destroys any preexisting agglomerates. Convection is the mixing process least likely to result in pronounced segregation caused by difference in size or other physical properties. Convective mixers typically transfer relatively large amounts of material throughout the mixing vessel, allowing only limited opportunities for particles to segregate. This is in contrast to tumbling blenders, where the material has many opportunities to segregate during the tumbling/avalanching phase of the mixing cycle. In addition, mixing occurs throughout the chamber in a convective blender, whereas in a tumbling blender, mixing is typically confined to the surface of the powder bed, leaving large regions undisturbed during long periods of the mixing cycle.

While providing resistance to segregation and agglomeration, the mixing mechanisms employed by a convective blender can produce potentially adverse consequences. The motion of the impeller induce high levels or rates of shear, which may damage the material being mixed. Two other effects that may occur in a convective blender are attrition (grinding the powder into finer particles) or intense heating of the powder. Attrition is a common phenomenon in these blenders, as the shearing action of the impeller blades can cause rapid reduction in the individual particle size. This effect is sometimes desirable, for example, when designing a process to equalize the particle size of the material being processed. The motion of the impellers may aerate the mixture and cause the regions near the surface to fluidize. Convective blenders are typically operated at rotational speeds (15 to 60 rpm) and fill levels (>50%) similar to those of tumbling blenders. However, no systematic study of mixing performance in terms of mixing time, rotation rate, fill level, or loading patterns has been published to date for any convective blender.

15-4.2.2 Applications and Types. The design of convective blenders allows for the efficient processing of a wide range of material states, including powders, granular solids, slurries, liquids, pastes, and combinations of these. Consequently, these blenders are utilized in a wide range of industries, including construction, agriculture, chemicals, pharmaceuticals, and foods. Convective blenders come in a wide variety of designs, all following the basic design of a stationary vessel swept by a rotating impeller. The stationary mixing chamber may be conical, cylindrical, or trough-shaped, and impeller designs range from ribbons to paddles to screws. The impeller may sweep though the entire mixing chamber each rotation, or it may stir small regions progressively until the entire chamber is stirred. Common convective blenders include the ribbon blender (a cylindrical vessel with a helical ribbon impeller mounted to a horizontal shaft), the paddle blender (a modified ribbon blender with paddles instead of a helical ribbon), and the Nauta blender (a vertically oriented conical tank swept out by a rotating and precessing screw impeller). Other blenders used in industrial applications include the Forberg mixer (two paddle blender drives sweeping two connected troughs), the Z-blade blender (a cylindrical vessel swept out by a Z-shaped blade), and the Lodige (similar to a kitchen mixer, where plough-shaped shovels rotate within a cylindrical drum). Further discussion of these mixers is provided in Section 15-10.3.2.

15-4.2.3 Ribbon Blender. The ribbon blender is one of the most common general purpose mixers, as it is capable of effectively performing a wide range of mixing processes including liquid, solid, and liquid–solid blending. Common industrial applications of these blenders include mixing the powder components of pharmaceutical tablets, blending oils and shortenings into dry ingredients to form a cake batter, and combining gravel and asphalt. A batch ribbon blender is depicted in Figure 15-23. The motion of the ribbon blades near the vessel walls can result in *pinch points*, regions of high shear and compression which may damage fragile materials or cause attrition. The capacity of these blenders is set by the span of the ribbon, which must clear the top of the powder bed in order to mix the entire bed. As is true for many convective blenders, the intensity of shear can result in heating that can adversely affect the quality of the product.

During operation of a standard ribbon blender, two sets of helical ribbon blades transport material in opposite directions; the outer ribbons will transport material toward the center of the mixing vessel while the inner ribbons transport material toward the ends of the vessel (Figure 15-24*a*). Turbulent convective currents caused by these counterrotating elements act to blend the different components. Unlike many tumbling blenders, a ribbon blender is often not completely discharged by gravity, requiring additional blade rotation to complete this process. This can result in additional mixing, segregation, and attrition, which must be taken into account during process design.

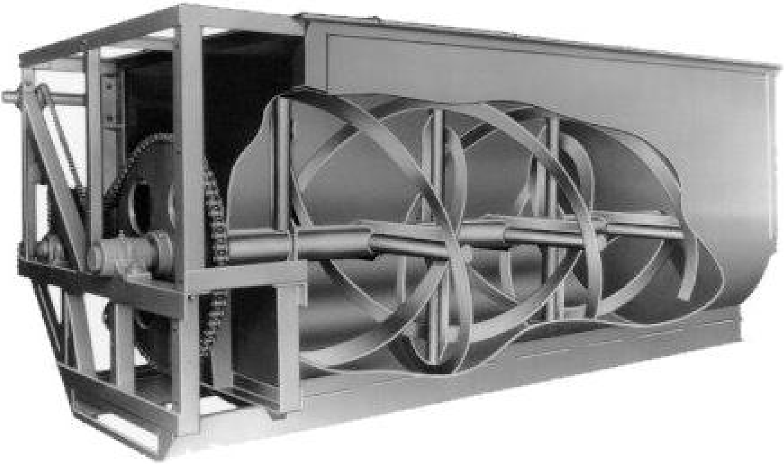
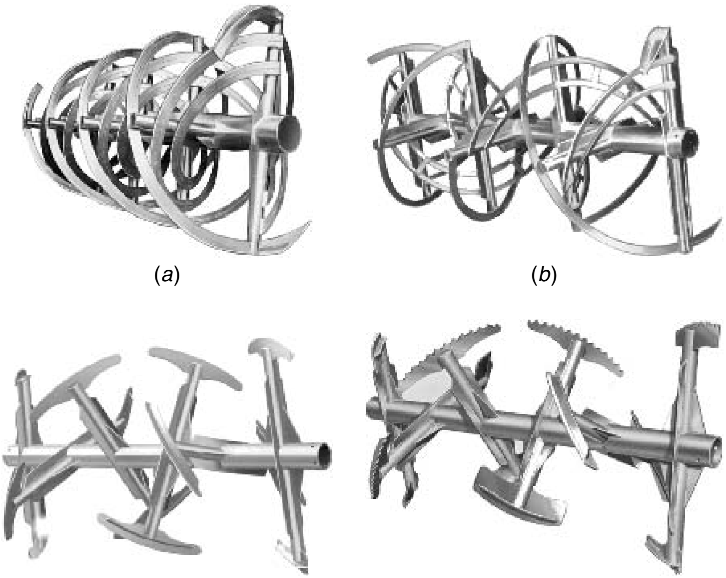


Figure 15-23 Schematic of a ribbon blender, consisting of a cylindrical vessel swept out by a rotating helical impeller blade. (Courtesy of H.C. Davis Sons Manufacturing Co., Inc., Bonner Springs, KS, printed with permission.)



(*a*) (*b*)

(*c*) (*d*)

Figure 15-24 Four convective blender impellers: (*a*) double ribbon agitator; (*b*) center-discharge ribbon agitator; (*c*) paddle agitator; (*d*) sawtooth paddle agitator. (Courtesy of H.C. Davis Sons Manufacturing Co., Inc., Bonner Springs, KS, printed with permission.)

Ribbon blenders are simple to modify for specific processes, and many refinements have been commercialized. The most common type of modification is to change the ribbon blade design; for example, Figure 15-24 shows some variations of the standard ribbon blade (Figure 15-24*a*). Figure 15-24*b* shows a common variation: the center-discharge ribbon. Here the two outer ribbons bring material to the center of the blender, while the inner ribbons force the ingredients outward to each end of the vessel. The paddle agitator (Figure 15-24*c*) contains both forward and reversing paddles in place of the smooth ribbon, constantly moving ingredients from one end to the other. To limit pinch points between the paddles and the blender surface, notches, or saw teeth, can be cut into the paddles (Figure 15-24*d*). Other types of modifications include creating a hybrid ribbon-paddle agitator or adding components to or removing sections of a ribbon to vary shear effects. Two agitators of the latter type are the cut-it-in ribbon agitator (a standard ribbon supplemented with cutting wires mounted on ribbon arms) used to cut thick materials (fats, oils, shortening) into powders (flour) and the cutout agitator (alternating sections of a standard ribbon are removed) used for heavier materials. Although there are many variations on the simple ribbon, these are all ad hoc, and there are few rigorous and usable scientific studies of these devices. See Section 15-10.3.2.3 for applications.

15-5 SELECTION AND SCALE-UP OF SOLIDS BATCH MIXING EQUIPMENT

As mentioned above, tumbling blenders can be grouped into two categories: convective blenders and tumbling blenders. Convective blenders rely on the action of impellers or paddles within a bowl, trough, cone, and so on, to move powders around and to generate a well-mixed product. Tumbling blenders consist of a hollow vessel attached to a rotating shaft; different blender types are identified by the geometry of the vessel. In either case, powders are mixed by rotating the blades or the vessel itself at a fixed rotation rate.

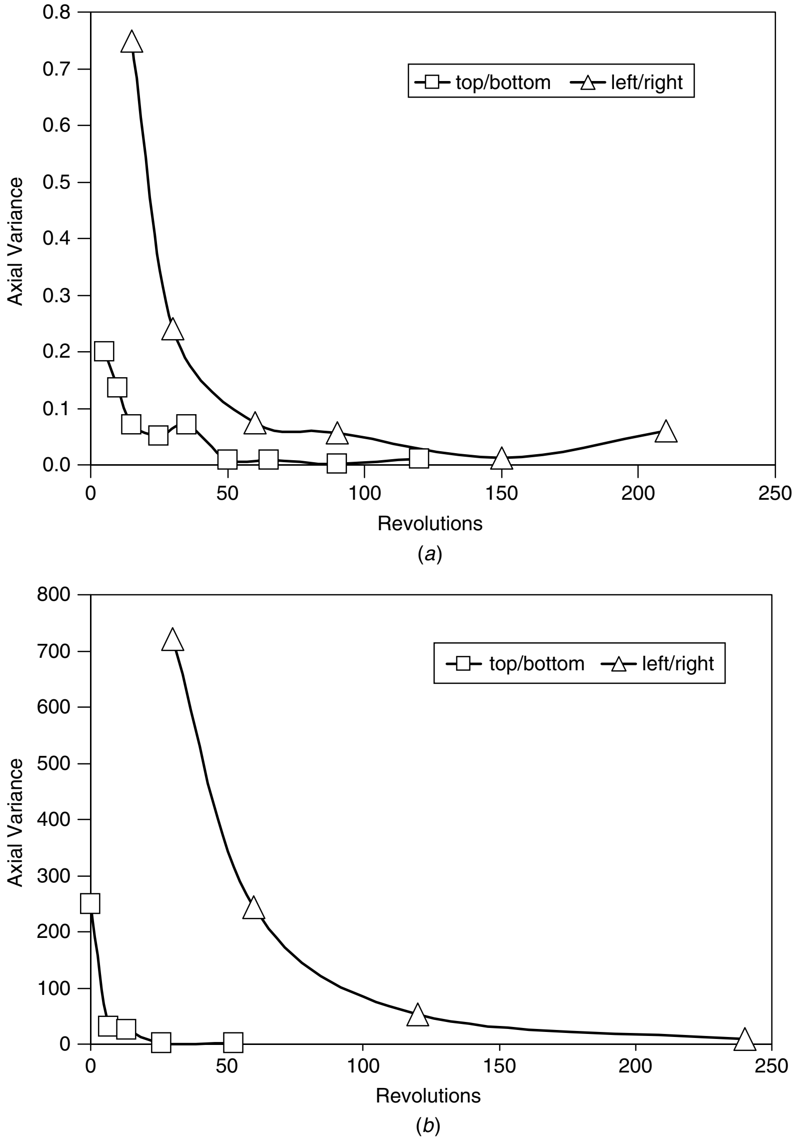
From a design and scale-up viewpoint, the major difference between the two types of blenders is the amount of shear imparted to the mixture during the blending process. In the absence of intensifier bars, tumbling blenders provide low-shear environments and are used when materials are shear sensitive or nonagglomerating. Convective blenders impart much more shear into the mixture and tend to be utilized for cohesive materials. Some tumbling blenders are equipped with a high-speed impeller, which can greatly increase the shear environment and allow for blending of some cohesive mixtures. From a manufacturing standpoint, tumbling blenders are often preferred because they come in a wide range of capacities and have shorter cleaning times. The choice of mixer often comes down to the properties of the mixture in question. Unfortunately, without well-established methods for measuring cohesion or agglomerating tendencies for different mixtures, it is impossible to develop a priori rules for blender choice based on the characteristics of the mixture materials.

In the remainder of this section we discuss recent findings regarding the scale-up of tumbling blenders, which have more easily classified flow fields and mixing mechanisms than convective blenders. As mentioned previously, the description of mixing mechanisms in convective blenders has not been the subject of considerable experimental investigation work, relegating scale-up considerations to trial and error.

15-5.1 Scaling Rules for Tumbling Blenders

The ensuing discussion will revolve around experiments run in 14, 56, and 300 L tote blenders using two mixtures: a free-flowing binary 50 : 50 w/w% mixture of 400 μm sand particles and a cohesive mixture of 3% micronized acetaminophen (∼30 μm) in a 50 : 50 w/w% matrix of PH102 Avicel and Fast-Flo lactose. All experiments were run at 60% of blender capacity at a rotation rate of 10 rpm. In raw form the acetaminophen was extremely cohesive and agglomerates (up to **O**(1 cm) in diameter) formed in the bulk mixture. The effect of blender scale on the breakup of these agglomerates is an important consideration for scale-up of tumbling blenders.

Probably the most important rule governing the basic dynamics of mixing in tumbling blenders is that the loading protocol has the most direct impact on the mixing rate. When the blender is symmetrically loaded in a top/bottom fashion,



(*a*)

(*b*)

Figure 15-25 Axial variance decrease for top/bottom- and left/right-loaded experiments in a 56 L tote blender with (*a*) a cohesive mixture and (*b*) a free-flowing mixture.

mixing rates can be more than an order of magnitude greater than when there are axial differences in the loading conditions. Figure 15-25 shows the decrease in axial variance (radial variance decrease, not shown, was nearly identical for both cases) for top/bottom- and left/right-loaded experiments using both the cohesive mixture and the free-flowing mixture.

Radial mixing rates (emphasized by top/bottom loading) are faster than axial mixing rates (emphasized by left/right loading) for both mixtures. However, the change in loading pattern has a much greater impact on mixing rates for the free-flowing mixture than the cohesive mixture. This disparity indicates that mixing mechanisms do not scale equivalently for the two mixtures, which can be a major consideration when changing vessel sizes.

15-5.1.1 Scale-up of Axial and Radial Mixing Rates. The effects of changing the vessel size on mixing rate is the predominant concern for the scale-up of manufacturing processes. Figures 15-26 and 15-27 compare the effect of changing blender size on the radial mixing rate (i.e., loaded top/bottom) and axial mixing rate (i.e., loaded left/right) for both cohesive and free-flowing mixtures.

For radial mixing rates, the change in vessel size has almost no impact on the observed mixing rate, regardless of mixture characteristics (variability in the cohesive data is caused by the capture of agglomerates). On the other hand, axial mixing rates are significantly different for the free-flowing mixture but nearly identical for the cohesive mixture. These differences in the scaling of the axial mixing process can be attributed to the difference in the way that these two mixtures flow in a tumbling blender.

Generally, when a free-flowing mixture is rotated in a tumbling blender, there is a regular flow, characterized by a nearly flat axial surface (i.e., there is little variability in bed height perpendicular to the mean flow), and particles travel along path lines nearly perpendicular to the axis of rotation. Cohesive mixture flow displays completely different behavior: flow is characterized by a series of dislocations that mark the onset of flow for a discrete portion of the mixture at various locations. These failures occur at seemingly random positions in the mixture and contain variable amounts of material. Thus, the surface of the cohesive mixture is marked by many hills and valleys, and flow down the cascade is rarely straight or perpendicular to the axis of rotation. Flow of cohesive material has an inherent axial component that greatly enhances axial mixing of the powder. Hence, the width (capacity) of the vessel does not play a significant role affecting mixing rates for cohesive mixtures, in stark contrast to free-flowing materials that rely on dispersion as the major mechanism for axial mixing. In this case, vessel width plays a dominant role.

15-5.1.2 Shear Effects on Mixing. Free-flowing mixtures do not exhibit significant particle–particle interactions, and the presence of shear, or lack thereof, has little or no effect on the mixing rate. It has been shown that mixing of free-flowing particles in tumbling blenders is independent of rotation rate and only a function of fill level and number of revolutions. However, for cohesive mixtures, the presence of shear can have a major impact on the homogeneity of the final blend.

Shear effects are demonstrated in Figure 15-28, which shows the evolution of the sample mean (mean value of all retrieved samples) from the cohesive mixture for top/bottom-loaded experiments in 14, 56, and 300 L blenders. To

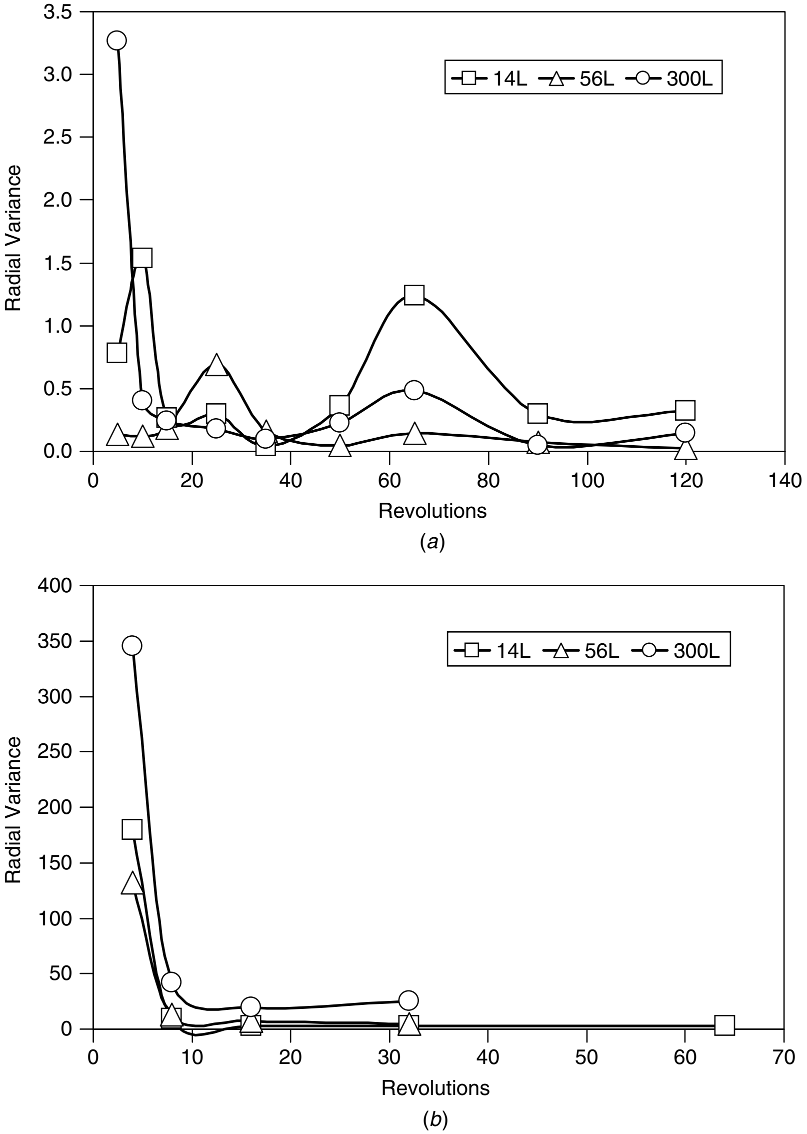


Figure 15-26 Radial mixing rates for top/bottom-loaded (*a*) cohesive and (*b*) free-flowing systems in tote blenders of 14, 56, and 300 L capacity.

highlight the effect of shear, the acetaminophen was loaded into the blender without presieving, so that numerous agglomerates were initially present in the mixture. Sample means for the smallest blender (14 L) reach a maximum of ~1.8% acetaminophen, indicating that a significant amount of acetaminophen was still trapped in clumps too large to be sampled. For larger vessels, the sample

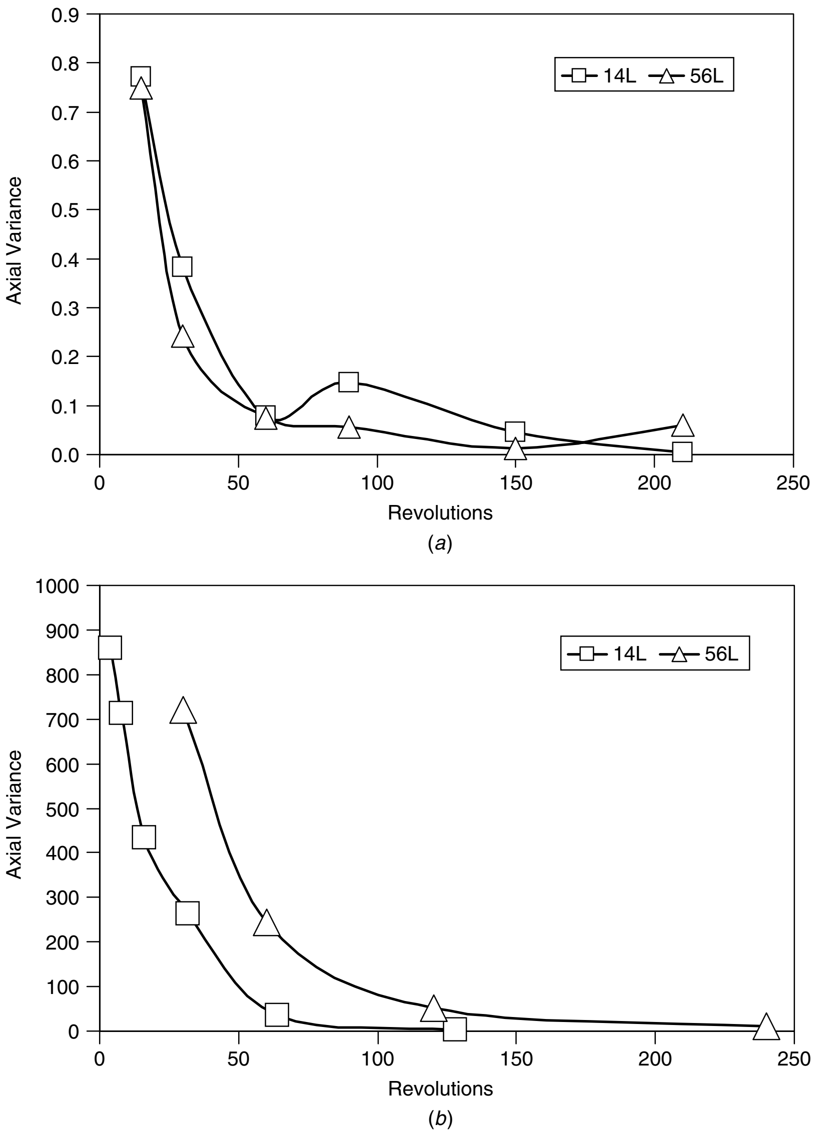


Figure 15-27 Axial mixing rates for left/right-loaded (*a*) cohesive and (*b*) free-flowing systems in tote blenders of 14 and 56 L capacity.

mean approached the mixture mean more rapidly; after 120 revolutions in the 300 L blender, the sample mean was 2.9%. The only mechanism for agglomerate breakup was through shear-induced diminution in the cascading layer.

All experiments were run at the same rotation rate, making it appear that shear energy scales with vessel size at constant rotation rate. Particle velocities in the flowing layer have been shown to scale with the radial length of the blender

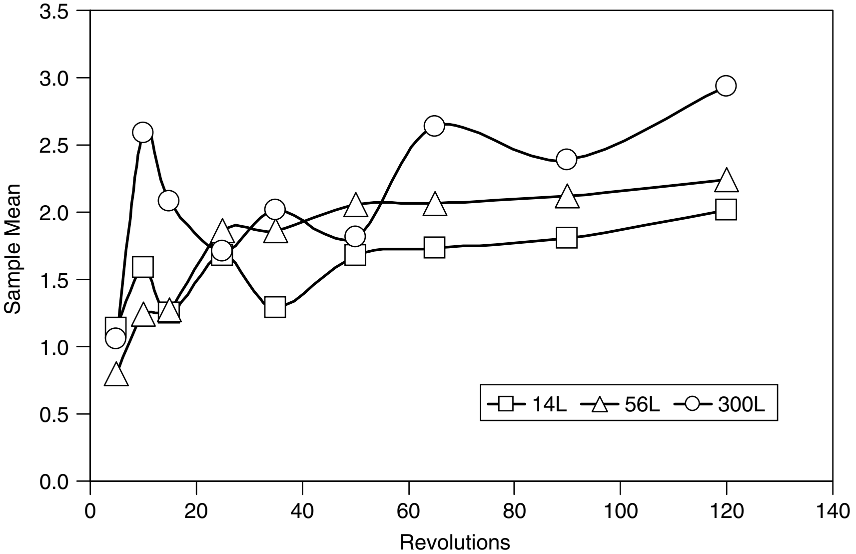


Figure 15-28 Evolution of the mean of all retrieved samples taken from 14, 56, and 300 L tote blenders. Larger blenders come closer to the actual mean value of 3%.

at constant rotation rate. Using particle velocity as a rough estimate for shear rates supports the idea that increased vessel size at constant rotation rates leads to increased shear and hence increased acetaminophen agglomerate dispersion into the bulk mixture. This finding can be enormously important for scale-down of mixing processes involving cohesive mixtures because agglomerate breakup, which is necessary for the achievement of a well-mixed final product, might occur more slowly (or not at all) in a smaller vessel. Shear energy can also be affected by changes in rotation rate, fill level, and the use of high-speed impellers. To frame scale-up rules properly for cohesive mixtures, it will be necessary to devise a means for quantifying changes in shear with these operational variables.

15-5.2 Final Scale-up and Scale-down Considerations

Generally, mixture characteristics (rather than blender characteristics) play the determining role in scale-up of tumbling blenders. For free-flowing mixtures, changing vessel size has an enormous impact on axial mixing rates, making the loading conditions the most important variable affecting the change in mixing rates with changes in scale. For cohesive mixtures, the amount of shear in the mixing process has the greatest effect on the mixing rate because the breakup and dispersion of agglomerates is necessary for creating well-mixed blends. Thus, one must be cautious of *increases* in vessel size for free-flowing mixtures, but for cohesive mixtures it is *decreases* in vessel size that pose the most problems. It is clear that the processes that control mixing in these devices will not be fully understood until quantitative methods for measuring cohesion of powder mixtures are developed.

15-6 CONCLUSIONS

Despite sustained efforts during the past decade both at Rutgers and elsewhere, powder blending remains largely an “art,” governed by empiricism and subject to frequent failure. In the opinion of the authors, the chief limitations in advancing the scientific understanding of powder mixing is a lack of effective experimental methods for measuring powder flow and powder constitutive behavior. Lacking hard data, constitutive modeling remains in its infancy, severely limiting our ability to achieve effective models for predicting powder flow and mixing from first principles. This situation is in stark contrast with the state of the art in fluid flow and mixing, which is discussed abundantly in the remainder of this book. For fluids, well-established methods for measuring constitutive behavior have greatly advanced our understanding of fluid rheology, which is a major building block of CFD models. Excellent EFD technology facilitates validation of CFD efforts. The net result is that fluid mixing systems can be designed with increasing reliability, and in many cases, entirely by computer. Although limitations exist, the fluids community is taking them by storm, one after another, at a rapid pace.

Thus, to advance beyond the current state of the art, we need to improve experimental techniques for measuring powder flow and powder constitutive behavior. Important efforts, mentioned earlier in this chapter, are under way using a variety of noninvasive technologies, but much remains to be done before powder blending processes can be designed and scaled-up reliably. The reader is advised to stay tuned, as the field is likely to evolve rapidly in coming years.

ACKNOWLEDGMENTS

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Many graduate students, postdoctoral researchers, and industrial collaborators have participated in this work in many ways over the years, and their contribution is acknowledged in the cited literature. The authors wish to express their special gratitude to dozens of Rutgers undergraduate students who tirelessly, and often anonymously, collected and analyzed the hundreds of thousands of samples that compose the data set and supports the observations and conclusions communicated in Part A of this chapter.

Part B: Mixing of Particulate Solids in the Process Industries

Konanur Manjunath, Shrikant Dhodapkar, and Karl Jacob

15-7 INTRODUCTION

Solid–solid mixing is a ubiquitous unit operation in particulate processes where consistency and homogeneity of the product is a key requirement. Mixtures are seen in all different phases of processing, ranging from the mixture of raw materials, as is the case with glass batch, ceramics, and blast furnace charges to the final formulation of products such as cereal mixtures, cosmetics, and polymer master batches. The quality of mixing is often key to product performance. For example, good mixing of cement and aggregate, color concentrate and base polymer, active ingredient in tablet formulations, individual components of a fertilizer formulation, and various components of a cereal mix is key to successful application.

Mixing can also be done in combination with other unit operations, such as agglomeration, size reduction, particle coating, and chemical reaction, to name a few. Often, selection of an appropriate unit operation (e.g., grinding, agglomeration) can reduce the cost of the process by combining two unit operations.

There is a wide variety of solids mixing equipment on the market today. Equipment ranges in size from small mixers that hold a few hundred pounds to large silos for large scale blending operations. It can be seen that certain mixers have long-standing domination of certain market segments, and improved designs have resulted from close cooperation with the end user. Newer concepts, such as Forberg mixers, continue to be introduced and accepted as the applications gain ground.

Selection of an appropriate mixer begins with an understanding of process requirements (e.g., quantity of bulk solids to be mixed, desired degree of mixedness, the need for batch integrity, upstream/downstream process) and material properties (e.g., particle size distribution, cohesiveness, particle shape, abrasiveness). The final analysis must then combine these factors with the operating and purchase cost to arrive at an engineering decision.

In this part of the chapter we cover solid–solid mixing as routinely practiced in the industrial environment, from small scale batch blending to continuous homogenization of polymer pellets. An effort is made to compile typical operating ranges and practical guidelines from various sources and the authors' experience. The following text is a summary of currently acceptable industrial practices; however, it is possible that certain segments of the industry may have variant practices. The focus here is also primarily on mixing of solids in the "dry" state. The subject of mixing/wetting of powders to form suspensions, slurries, and pastes is not treated here. A detailed discussion of tumbling mixers and a fundamental treatment of mixing phenomena was covered in detail in Part A of this chapter.

15-7.1 Scope of Solid–Solid Mixing Tasks

A myriad of different mixing tasks are performed in the process industries today. Some common tasks are:

- Mixing of product for homogenization of quality or reduction of variance (e.g., blending of polymer pellets, blending of batches in a lot)
- Mixing of active ingredient onto a carrier material (e.g., formulation of insecticides or herbicides for household applications where the carrier particles are clay granules or other inerts)
- Mixing of multicomponent mixtures as a formulation (e.g., cereal mix, specialty polymers)
- Coating of a cohesive component onto a carrier particle (e.g., coating of antiblocking agents on polymer pellets or granules, formulation of agricultural products)
- Mixing of fine powders to create a homogeneous mixture at the particulate level (e.g., masterbatch preparation for medicinal drugs)
- Coating of liquid additives onto the base material (e.g., waxes or additives on polymer granules, food applications)

The nature of application and process requirements will dictate the selection and specification of mixing equipment.

15-7.2 Key Process Questions

There are numerous factors that govern the successful mixing of bulk solids. Although it is difficult to make a complete and absolutely comprehensive list, there are several key process questions that deserve attention prior to mixer selection.

- What are the desired mixture quality and acceptable variation? This is a key process issue. It is extremely important to determine the acceptable mixture quality and variation early in the process of selection of a new mixer or while troubleshooting an existing mixer.
- What quantity of material is to be mixed or homogenized?
- What is the nature of the process? Is the process more conducive to batch or continuous mixing?
- Are there other unit operations, such as grinding, granulation, and drying, which can be combined with the mixing operation?
- What are the consequences of product degradation and cross-contamination? Does the equipment need to be cleaned frequently?
- Is the production rate constant? What kind of turn-up or turn-down is needed?

- Does the mixing process need to be contained because of industrial hygiene and safety concerns?
- What are the safety issues related to rotating piece of equipment or fire and explosion hazards?
- Does the process need to have on-line control? What is the extent of automation required?
- What are the constraints from space and accessibility perspective?
- What is the cost to benefit analysis?

15-8 MIXTURE CHARACTERIZATION AND SAMPLING

The ability of particles to mix and their tendency to segregate depend on differences in their size, density, shape, elastic properties, surface characteristics, and magnitude of interparticle forces. The difference in particle size is probably the most important factor. Unlike immiscible liquid systems, the density differences play a relatively minor role in de-mixing or segregation of particulate mixtures. The large body of literature available on fluid mixing therefore cannot be used to predict or evaluate solids mixing applications.

15-8.1 Type of Mixtures

Terminology associated with mixture classification is mired in controversy (Egermann, 1980; Thiel, 1982; Nienow et al., 1985; Hersey, 1975). Previously accepted terms have come under scrutiny as our understanding of mixing mechanisms has improved over the years. There is a serious lack of homogeneity and consistency in the terms used in the literature. An effort is made here to present basic concepts and various viewpoints on mixture classification.

15-8.1.1 Perfect Mixture. A perfect mixture of two types of particles is one in which any sample randomly taken from the mixture will contain the same proportion of each particle as the proportions present in the mixture taken as a whole. As shown in Figure 15-29*a*, alternate arrangement of black and white particles will create a perfect mixture. Such perfect mixtures are rarely found in nature.

15-8.1.2 Random or Stochastic Mixture. When two noninteracting components (e.g., free-flowing pellets) with similar properties (size, shape, elasticity, etc.) are mixed in an ideal mixer, the quality of mixing reaches an asymptotic limit of random mixing (see Figure 15-29*b*). This is a statistical process or probabilistic process. Each square in Figure 15-29*b* has a 50 : 50 chance of being black or white. Extended mixing of random mixtures does not result in improvement of mixing quality. For particles with different physical properties, it is not always possible to achieve a random mix. A random mixture cannot be achieved in the presence of significant interparticle forces (e.g., van der Waals, electrostatic, cohesive).

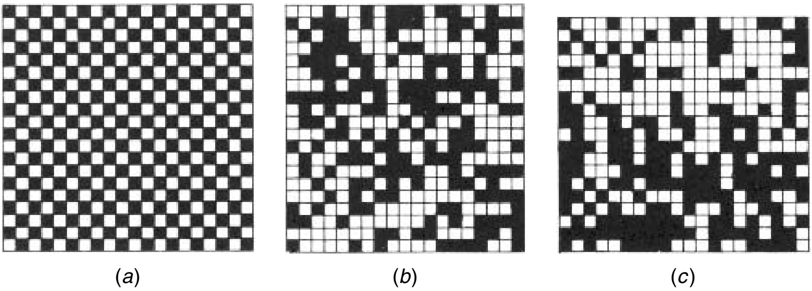


Figure 15-29 Simulated mixtures: (*a*) perfect mixture; (*b*) random mixture; (*c*) segregated mixture. (From Williams, 1986.)

15-8.1.3 Ordered Mixture. When two interacting components are mixed together, a structure or order can build up into the mixture. The units ordered could be a result of agglomeration or cohesion of one component to the other or a mixture of the two. A perfectly ordered mixture can be obtained by:

1. Applying sufficient energy to break any agglomerate of the cohesive fraction (minor component) and distributing it on available sites on the carrier particles. The available sites should be sufficient to hold the cohesive fraction; otherwise, the cohesive particles can reagglomerate.
2. Ensuring complete randomization of carrier particles.

It should be noted that an ordered mixture can have a variance less than that of a random mixture. A perfectly ordered mixture will have zero variance.

15-8.1.4 Partially Ordered Random Mixture. Once the cohesive fraction or minor component saturates available sites on the carrier particles, the remaining fraction will agglomerate. In partially ordered random mixtures, these agglomerates and the ordered units are randomly mixed together.

15-8.1.5 Pseudorandom Mixture. When the ordered units contain different number of adherent particles and the carrier particles are randomly mixed, the mixture is called a *pseudorandom mixture*. The carrier particles are not saturated with the minor component, and there are no agglomerates in the mixture.

The following illustration (Stainforth, 1982) in Figure 15-30 is an excellent summary of various types of mixtures and the influence of surface forces.

15-8.1.6 Alternative Definitions. Egermann (1980, and Table 15-1) proposed that the nomenclature reflect the degree of homogeneity of the mixture rather than the underlying mechanisms. He proposed the following definitions:

- *Ideally ordered or perfect mixtures* are defined by a standard deviation of the sample composition equal to zero provided that the sample size is greater than one ordered unit.

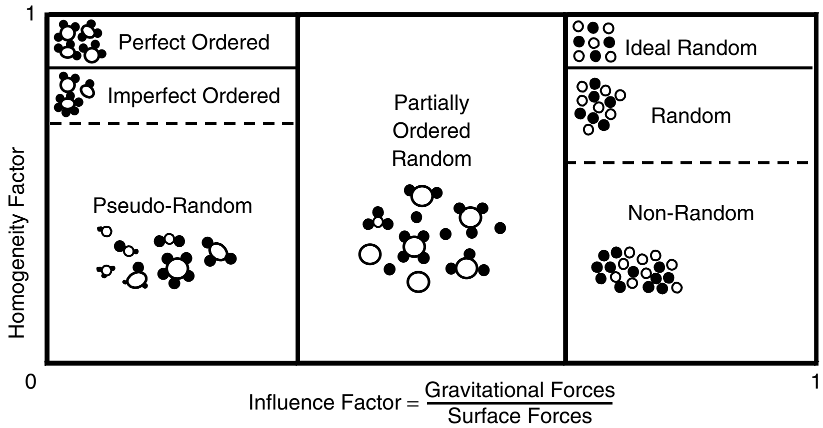


Figure 15-30 Representation of relationship between various mixtures. (From Stainforth, 1982.)

Table 15-1 Summary of Mixing Nomenclature

Type of Mixture	Homogeneity	Some Degree of Order?
Ideally ordered (perfect)	σ equal to 0	Yes
Ordered	σ smaller than σ_R	Yes
Pseudorandom	σ equal to σ_R	Yes
Random	σ equal to σ_R	No
Incomplete	σ higher than σ_R	Yes

Source: Egermann (1980).

- *Ordered mixtures* feature a higher degree of homogeneity than random mixtures. The standard deviation will be smaller than random standard deviation.
- *Random mixtures* show a completely disordered distribution of the individual particles in the absence of interparticle interactions.
- *Pseudorandom mixtures* show the degree of homogeneity but not the fully disordered texture of random mixtures.
- *Incomplete or segregated mixtures* are of poorer quality than random mixtures.

15-8.2 Statistics of Random Mixing

If random samples of N particles are taken from the known mixture of average composition q, where the fraction of the first component is P and second component is 1 – P, the composition of the samples will be normally distributed with a standard deviation of

$$\sigma = \sqrt{\frac{P(1-P)}{N}} \tag{15-6}$$

As can be seen, as the sample size decreases, the variance or standard deviation increases. In a mixture of total mass W, if m random samples are analyzed for concentration of component 1 (concentration $c_1, c_2, c_3, c_4, \ldots, c_m$), the sample mean can be calculated as

$$\overline{X} = \frac{\sum_{i=1}^{m} c_i}{m} \tag{15-7}$$

The standard deviation of a set of samples is given by

$$s = \sqrt{\frac{(c_i - \overline{X})^2}{(m-1)}} \tag{15-8}$$

It is assumed here that the sample concentrations are normally distributed about the mean.

It is known that multiple samples taken from a bulk (population) will give a distribution of measurements. For completely random mixture, such as a mixture of two free-flowing granular materials of equal size and density, the variance can be calculated theoretically as

$$\sigma^2_{\text{random mixture}} = \frac{P(1-P)}{N} \tag{15-9}$$

where P is the fraction of one of the components in the mixture and N is the number of individual particles.

For nonrandom mixtures, Williams (1986) found that the relationship above no longer holds. The relationship between sample size and variance is

$$\sigma^2_{\text{nonrandom mixture}} = \left[L + \frac{P(1-P) - L}{N}\right]^2 \tag{15-10}$$

Figure 15-31 shows the effect of sample size on the standard deviation for a nonrandom mixture. L is a constant for a given mixture or state of mixedness. It can be determined experimentally if the value of σ is known at one value of N. The condition of $L = 0$ corresponds to random mix:

$$\lim_{L \longrightarrow 0}[\sigma_{\text{nonrandom}}] = \sigma_{\text{random}} \tag{15-11}$$

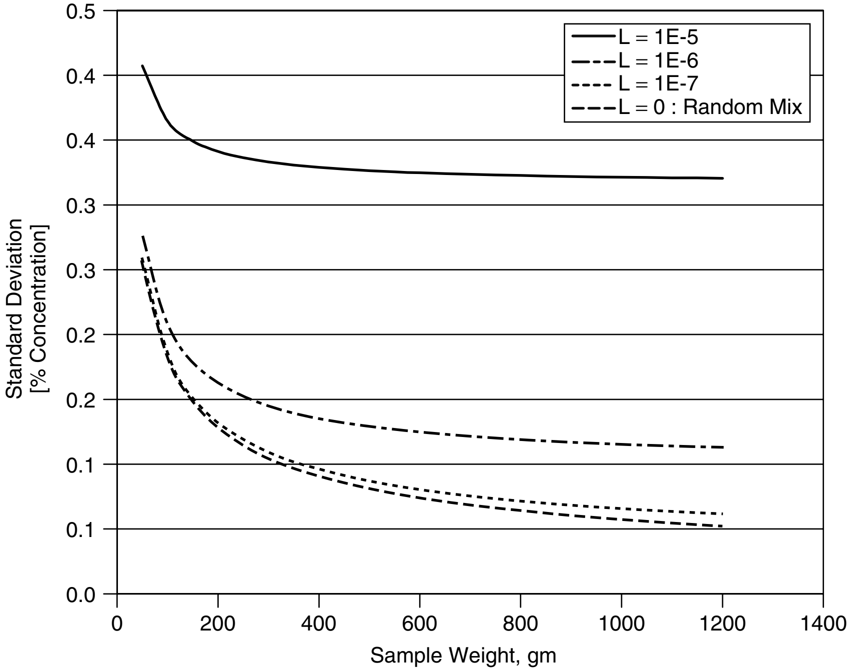


Figure 15-31 Effect of sample size on mixture standard deviation for nonrandom mixtures.

For a system where the two components are completely unmixed, the variance σ_o^2 of the sample composition is

$$\sigma_o^2 = P(1 - P) \tag{15-12}$$

A completely unmixed system can be visualized as filling a jar first with component 1 and then topping it with component 2 without disturbing the layer of component 1. Note that the expression above is independent of the sample size. It is also assumed here that no sample straddles the boundary between the two components.

The equations discussed so far assume that all the particles in the mixture have the same size. However, if we have a binary random mixture in which each component has a particle size distribution, it is necessary to calculate the number of particles of each component independently. Stange's (1954) derivation has been used widely:

$$\sigma_R = \sqrt{\frac{P(1 - P)}{W/[Pw_P(1 + C_P^2) + (1 - P)w_{1-P}(1 + C_{1-P}^2)]}} \tag{15-13}$$

where W the total weight of the sample, P is the mass fraction of the first component corresponding to σ_R, w_P the weight fraction of the first component, w_{1-P} the weight fraction of the second component, and C_P the coefficient of variation of the particle weight of the first component, C_{1-P} the coefficient of variation of the particle weight of the second component.

15-8.3 Interpretation of Measured Variance

The total measured variance for a set of samples taken from bulk (population) is summation of contributions from various sources: namely,

$$\sigma^2_{\text{measured}} = \sigma^2_{\text{mixture}} + \sigma^2_{\text{sampling}} + \sigma^2_{\text{analytical}} \qquad (15\text{-}14)$$

The sampling and analytical variances must be determined from independent measurements. It is well established that once the analytical uncertainty (standard deviation) is reduced to a third or less of the sampling uncertainty, further reduction in analytical uncertainty is of little importance. Therefore, if the uncertainty in sampling is very large, it may be beneficial to opt for an analytical method that is rapid even though it might have lower precision. This will permit more samples to be analyzed, thereby resulting in a better estimate of the mean value.

15-8.4 Sampling

To evaluate the state of mixedness of a mixture, a representative sample must be retrieved and analyzed. The result of this analysis combines errors due to sampling, analytical method, and uncertainty due to state of the mixture. Following certain guidelines and good practices can minimize the error due to sampling and analysis. The subject of sampling is very complex and detailed treatment can be found in the literature (Hersey, 1970; Kristensen, 1973; Sommer, 1986; Muzzio et al., 1997). The objective of this section is to outline important issues and provide practical guidance.

Some common questions pertaining to sampling encountered during mixer selection and performance evaluation are:

- Where should the samples be obtained?
- How should the optimal sample size be determined?
- How often should samples be collected?
- What apparatus is available for sampling from stationary material and from moving (process) streams? How should the appropriate unit be selected?
- How should the gross sample be reduced to analytical size with minimal bias?

15-8.4.1 Sampling Location. The selection of sampling locations depends on the objective of the study and mode of mixer operation. The objective of the mixing study could be (1) selection of a mixer for a given process, or (2) evaluation of the performance of mixer in the process. A mixer could be operated in batch or continuous mode.

For selection of a mixer, it is important to understand the spatial and temporal variability of the characteristic property within the mixer. The entire volume of the mixer must then be spatially divided and samples randomly taken. Knowledge of flow patterns within the mixer is helpful to identify the location where material is likely to be stagnant.

For performance evaluation, it is recommended that samples be taken at the discharge spout. From a process perspective, it is important that the mixture coming out of the mixer is homogeneous and no segregation occurs during discharge. It is possible for a well-mixed sample to de-mix during discharge if proper care is not taken. Proper care implies that one is cognizant of the segregation mechanisms possible for the mixture and implementation of proper engineering controls to control it.

One must follow the two *Golden rules of sampling* as proposed by Allen (1981):

1. Sample a moving stream.
2. The whole of the stream should be taken for many short increments of time in preference to part of the stream being for the whole of the time.

15-8.4.2 Selection of Sample Size. Ideally, the size of a sample should be equal to the scale of scrutiny of the mixture. The scale of scrutiny is the scale (or amount) of material at which homogeneity is desired. For instance, for certain pharmaceutical applications, the size of a single tablet is the scale of scrutiny where it is critical to ensure that the active ingredient is well mixed to the level of single tablet. Similarly, for polymer extrusion processes, the scale of scrutiny is the volume of the polymer mixing zone in the extruder. For agricultural chemicals, one or a number of bags of fertilizer could be considered as the appropriate scale. Determination of scale is independent of the property that is being scrutinized.

When it is not possible to obtain samples comparable to the ideal size, appropriate sample size reduction techniques must be employed to obtain representative sample for analysis. For random mixtures, the sample variance is inversely proportional to the sample size. For a certain composition of noninteracting particulate and a given sample size, there exists a minimum theoretical value of standard deviation that can be achieved through random mixing. If the chosen sample size is very small, one must take a large number of samples to reduce the uncertainty in determination of mean mixture property (Student's t-test).

15-8.4.3 Number of Samples or Sampling Frequency. The value of sample variance approaches mixture variance as the number of samples becomes very large (following chi-square statistics). For batch mixers it is common practice to stop the mixer and sample the stationary bed at various locations. Since the mixture mean or standard deviation is not known a priori, historical data must be used as guideline. In continuous mixers, a sample can be obtained at the mixer outlet following the golden rules of sampling. Care must be taken to avoid any long-term cycling of the process variables. In practice, the number of samples, is limited by the capability of the analytical technique to process the samples. For online measurement devices, such as those that measure moisture, higher sampling frequency can be implemented.

15-8.4.4 Samplers and Their Selection. Although it is highly recommended that the golden rules of sampling be followed, sampling situations from a stationary bed or pile are unavoidable. If the total quantity of a stationary bed or pile is not very large, a chute riffler or spinning riffler should be used to obtain a representative sample. If the stationary bed is large or while sampling from a mixer, one may use a sampling thief, pneumatic lance, or a scoop. Each of these methods results in biased sample and must be used with caution (Allen, 1981). See also Section 15-2.2.

As mentioned earlier, it is best practice to sample a mixer at the discharge location. A number of online samplers are available in the market: whole stream samplers, cross-cut samplers, and split-stream samplers. The following factors must be considered for selection of an appropriate sampling device:

1. *Flowability of the material* (cohesive versus free-flowing). The sample must be capable of flowing into the sampler.
2. *Maximum particle size*. The sampler must be sufficiently large to accommodate the maximum particle size.
3. *Friability of the material*. The sample must not be crushed in the sampler.
4. *Size of sample desired*. The sample size should match its intended use.
5. *Availability of space*. Because of their large size, some samplers cannot be fit into an existing process.

15-8.4.5 Sample Size Reduction. The sample obtained from the sampler is generally larger than the sample required for analysis. If a small portion of this sample is arbitrarily scooped for analysis, it will introduce a bias. The sample size can be reduced to the analytical size using various sample reduction techniques: (1) spinning riffler, (2) chute riffler, (3) ICI method, and so on. All these methods follow the golden rules for sampling. For details on these sample reduction techniques, see Allen (1981).

15-9 SELECTION OF BATCH AND CONTINUOUS MIXERS

In this section the distinction between batch and continuous mixers is discussed so that appropriate selection can be made to suit both the process and mixture requirements [see also Brennen (1990) and Michael (1992)].

15-9.1 Batch Mixing

Batch mixing is mixing ingredients in any amount in individual batches in an individual mixer or a vessel. All ingredients are loaded into a mixer and agitated for a certain period until they are homogeneously distributed or mixed. The resulting mixture is then discharged out of the vessel. The critical parameters that influence the selection of such mixers is the mixing duration, the size and the geometry of the mixer, and the operating conditions.

15-9.2 Continuous Mixing

Continuous mixing is used to mix ingredients continuously in a mixer in a single pass. The ingredient quantity to be mixed may vary in any range; however, unlike batch mixing, care must be taken to feed the mixer in a controlled fashion. Mixing in batch often leads to variation in the mixing quality, which can be controlled or almost eliminated by continuous mixers. Even though continuous mixing is gaining popularity, selection of continuous mixers is not as straightforward as batch, which can be accomplished by running trials.

Continuous mixers are more compact than batch mixers. While discharging from the mixers, segregation can be reduced by positioning the discharge closer to packaging units or as an integral part of it. In continuous mixing, mixing has to be achieved in both the radial and axial (in the direction of conveying) directions.

15-9.3 Comparison between Batch and Continuous Mixing

Advantages of Batch Mixing

- Any type of powders either cohesive or free flowing can be well mixed.
- Loading either powder or liquid ingredients into the mixer is straightforward.
- Mixers are easier to maintain and clean.
- It is easier to identify a batch for further follow-up (batch integrity).

Disadvantages of Batch Mixing

- It is not suited for minor ingredients, particularly at very low loading, since the ingredient can get coated onto the vessel.
- Segregation is unavoidable and may be severe if the packaging is located far from the discharge.
- If changing ingredients, the mixer must be cleaned after every batch, work that is labor intensive.

Advantages of Continuous Mixing

- *High capacity.* Compared to batch types, continuous mixers of small capacity and power can be used to produce large quantities of a mixture. Hence, for a given capacity, they are more compact than batch mixers.
- *Efficient dispersion of minor ingredients.* Mixing is very intense and thorough. Minor ingredient can be mixed more effectively.
- *Low hold-up.* The residence time in continuous mixers is much less and hence the holdup in continuous mixers is usually low.
- *Suitability for automatic control.* These systems are suitable for the application of online instrumentation and quality control. It is also possible to carry out formulation and rate changes to match the performance to the process requirements.
- *Minimum segregation.* Continuous mixing can cut down and control segregation of products because it can be located physically close to the next unit operation. If a batch mixer is selected for such a case, one could expect segregation during discharge of the product from the blender and subsequent handling of the mixture.
- *Low cost.* Continuous mixers tend to be cheaper than the equivalent batch mixer because they are compact and require less space. However, the cost for the feeders for metering the product into the mixer could be higher, especially if the number of components being mixed is large.
- *Minimum labor.* Since filling and emptying goes on automatically, minimal labor is required.

Disadvantages of Continuous Mixing

- *Lack of flexibility.* The continuous mixer is designed for a particular application and it cannot easily be tailored to mix a number of different ingredients unless the necessary facilities are built into the system at the outset. The effective turndown for batch mixer is quite high, and it is easier to vary the production rate. Continuous mixers generally have feeders that are precisely controlled, and it is not easy to change these narrow limits around the feed rate. If a new ingredient is introduced, this calls for a change in the protocol, and the feeders have to be calibrated to suit the application in hand.
- *Equipment break down.* Continuous mixers depend on many other pieces of equipment, such as the metering feeders and the monitoring devices to function at the level desired. If any piece of equipment fails to operate reliably, the entire mixing process is affected. Once the feeders are set to deliver the desired mixture quality, the system must be left undisturbed except for the purpose of calibration.

- *Calibration and checking*. The feeding devices require careful calibration, particularly if the process is being operated within a narrow range. To have good mixture quality, all associated equipment must be properly maintained.
- *Batch integrity*. This is something well suited with batch mixing operations. In continuous mixing, bad batches of incoming products get mixed, and it can be very difficult to spot the "off-specification" product.
- *Good sampling procedures*. To keep track of the mixture quality, one has to have an inspection routine for the mixture and for the metered feeds to the mixer. Variation in the input quality and quantity will affect the final mix.
- *Component limitation*. Since the final mixture is a function of the feed products, one will have to specify feeds in a very tight range, and this will increase the cost of the final mixture. If multiple components must be fed, batch is mostly preferred.
- *Controlled feeding*. Controlled feeding of input materials is required, and this will increase the capital costs for such mixers. The throughput is more or less fixed and is difficult to vary without significant modification of the expensive feeding and controlling devices.

15-9.4 Selection of Mixers

15-9.4.1 Selection of Batch Mixers. A selection chart for batch mixers is shown in Figure 15-32. The chart, however, excludes consideration of properties such as abrasiveness of the product, the buildup of static charge in the mixer, friability of ingredients, and so on.

15-9.4.2 Selection of Continuous Mixers. A selection chart is shown in Figure 15-33 for deciding whether batch or continuous mixers are suitable, based on criteria proposed by van den Bergh.

15-10 FUNDAMENTALS AND MECHANICS OF MIXER OPERATION

See also discussions by Bridgewater (1976), Williams (1986), Fan et al. (1990), and Harnby et al. (1992).

15-10.1 Mixing Mechanisms

There are three underlying mechanisms for solids–solids mixing: diffusive mixing, shear mixing, and convective mixing. Diffusive mixing occurs due to small scale random motion of particles when they roll over a free surface. A high degree of particle mobility is required. When the material is sheared, either externally with a mechanical agitator or internally due to rotating motion of the shell, the shear zones within the bulk cause mixing by exchange of particles across the shear zones. Large scale mixing or convective mixing can be achieved by moving large portions of material from one location to the other within the bulk. The particles do not have much mobility, and therefore segregation is minimized.

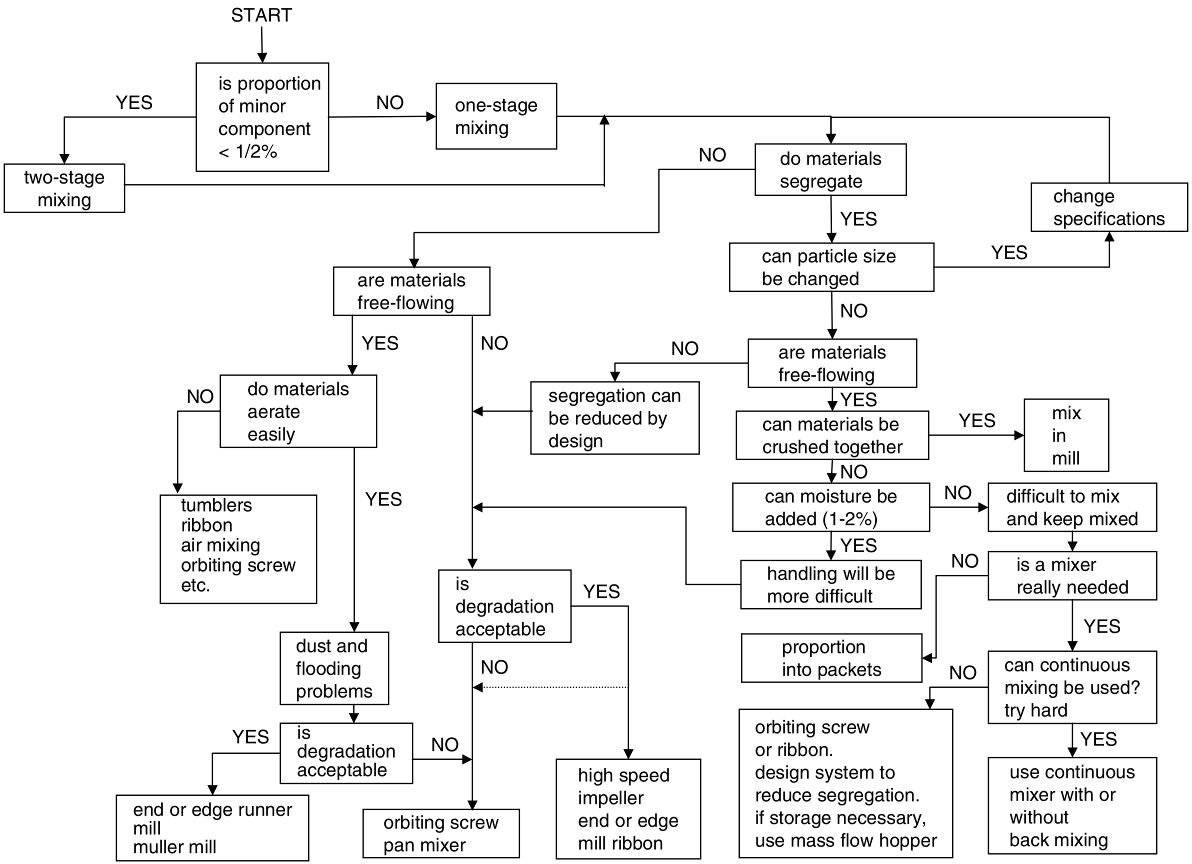


Figure 15-32 Selection chart for a batch mixer. (Modified from Miles and Schofield, 1970a.)

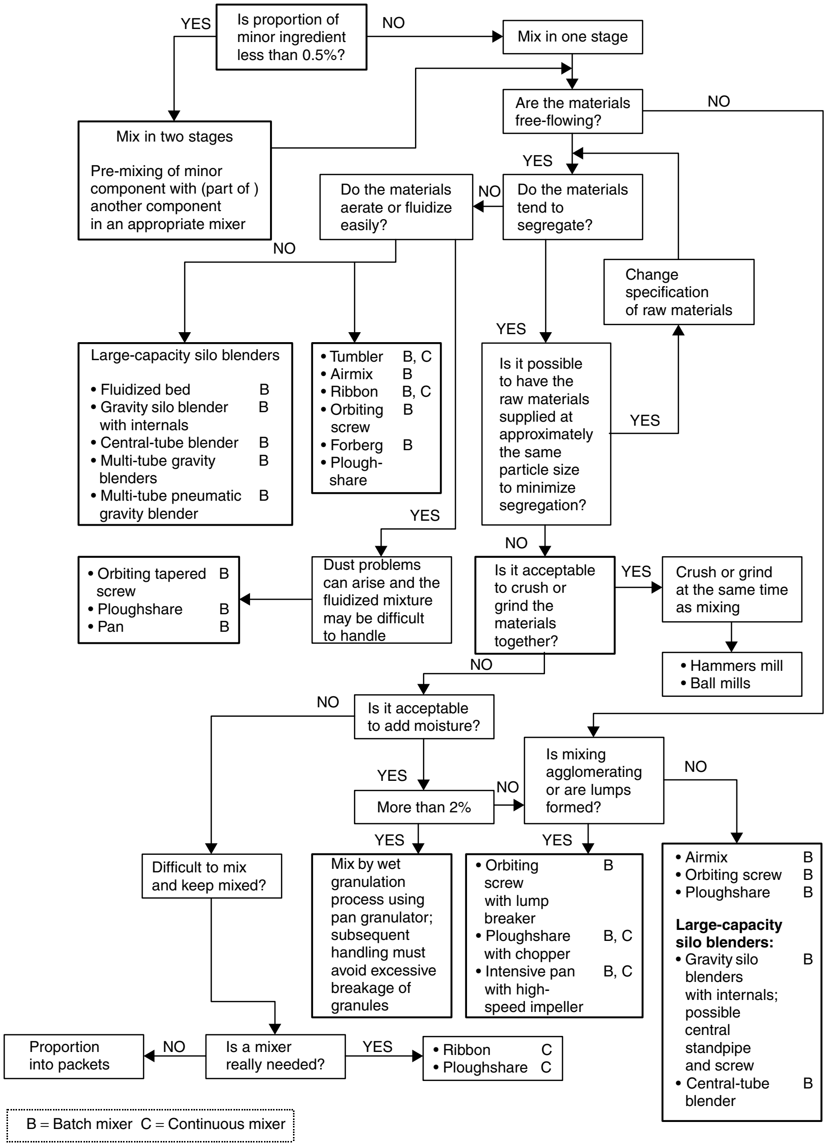


Figure 15-33 Selection criteria for continuous and batch mixers. (Modified from van den Bergh, 1994.)

During mixing, irrespective of the type of mixers, all the three mechanisms will occur, and one will probably dominate. Some of the mixing mechanisms can also result in segregation. For instance, free motion of particles on the surface (diffusive mixing) can also result in size-based segregation. Extended mixing time may actually cause de-mixing and poor mixture quality. Therefore, an understanding of the potential segregation mechanisms is helpful while selecting a mixer. Similarly, a low-shear tumbling mixer can cause agglomeration of a fine fraction in a fine/coarse mixture.

15-10.2 Segregation Mechanisms

No discussion of process mixing can take place without a concurrent discussion on segregation of particles. It is often said that segregation (particularly in batch mixing processes) begins the moment the agitation is switched off! Since the process engineer/operator has typically taken great care to mix the components, segregation is usually an undesirable side effect that must be recognized, and either appropriate engineering controls must be installed or the mixture must be "designed" such that it cannot segregate.

There are five generally recognized mechanisms of segregation: momentum, trajectory, sifting, fluidization, and air currents. These are most significantly affected by one particle property, particle size. Although there are other mechanisms involving other particle properties (e.g., the authors have witnessed segregation induced by a difference in coefficient of restitution), these five predominate and are described below (Carson et al., 1986).

15-10.2.1 Momentum. This mechanism occurs when a flowing bulk solid impacts a pile that is formed below the bulk solid. Because of differences in the momentum and in some cases the cohesion between the coarse and fine fractions, the coarse particles tend to gather at the outer periphery of the pile while the fines are deposited directly below the point of impact. This mechanism is quite common and has been shown to occur when the ratio of particle diameters is as small as 1.3. This mechanism would probably occur when, for example, a batch mixer is discharged into a hopper below it. Fortunately, the use of mass flow hoppers can effectively combat this radial segregation.

15-10.2.2 Trajectory. When a stream of bulk solids is allowed to flow off a conveyor or an inclined chute, segregation of the coarse and fine fractions can occur. The fines tend to drop directly below the chute/conveyor, while the coarse fraction is flung away. In mixing operations, care must be exercised when discharging either a batch or continuous mixer through an inclined chute. If this chute empties into a hopper, mass flow hoppers can help to combat the segregation, but not as effectively as the case for radial segregation. This is because of the asymmetric radial distribution of sizes in the hopper. If the mixture is to be packaged directly upon emptying the mixer, it may be possible for segregation in the package.

15-10.2.3 Sifting. This mechanism occurs when fine particles sift (much like a screening operation) into the interstices between the coarse fraction. One common cause is the shearing flow of bulk solid, as in the case of emptying a funnel flow silo (centrally moving core of material with a nonflowing annular region).

Here fine particles sift in between the coarse as the bulk solid avalanches down toward the center of the silo. This usually manifests itself by showing an increase in fines content in the material exiting the silo at the end of the discharge of the silo. Another common cause of sifting is vibration. In this case, the finer fraction can sift into the interstices of the coarse. This occurs frequently during product shipment.

15-10.2.4 Fluidization. When conveying material into a silo, it is not unusual for the fines to remain suspended in the headspace above the material for a long period of time, eventually settling as a layer on the material conveyed. This mechanism is likely to occur when mixtures containing slow settling particles (usually less than 100 μm) are conveyed.

15-10.2.5 Air Currents. Air currents within a vessel can cause the fines and coarse to segregate. This is quite common when a polydisperse mixture is being conveyed into a silo or hopper. The resulting segregation pattern is difficult to predict, but since the powder is being spread across the entire cross-section of the silo, mass flow can aid in remixing the segregated material.

15-10.3 Mixer Classification

Most industrial mixers can be broadly classified into the following categories:

- Tumbling mixers (V-cone, double cone, etc.)
- Agitated mixers
  - Paddle and plow mixers
  - Ribbon mixers (vertical and horizontal)
  - Screw mixers (vertical and horizontal and orbiting types)
  - Sigma-blade and Z-blade mixer
  - Forberg mixer
- Gravity silo blenders
- Pneumatic blenders
- High intensity mixers
  - Henschel mixer
  - Paddle mixer
- High-intimacy or high-shear mixer
  - Muller mixer
  - Compaction rollers

A brief description, typical operating ranges, and practical application information for these mixers follow.

15-10.3.1 Tumbling Mixers. As the name suggests, these mixers "tumble" the powder mass. Mixing is achieved predominantly by random motion when particles roll down a sloping surface. As the whole shell tumbles either on its own axis or eccentrically, the ingredients are bodily displaced, and mixing takes place in the radial direction. To achieve better dispersion, internal baffles and/or counterrotating impellers can be mounted and can be tailored for difficult-to-handle mixtures as well. Stationary spray nozzles for liquid coating can also be mounted along the axis of rotation.

Typical tumbling mixers are:

- Double-cone blender (batch); refer to Figure 15-19
- V and Y blenders (batch); refer to Figures 15-19 and 15-20
- Cylindrical blender (batch or continuous)

Tumbling mixers are discussed in detail in Section 15-4.1.

15-10.3.2 Agitated Mixers. Agitating mixers [see also discussions by Pahl (1986), Steiss (1995), Fuller (1998), Kent (2002), and Ramponi et al. (2002); discussion of convective mixers in Section 15-4.2] use mechanical means (e.g., paddles, plows, and ribbons) to create mixing action while keeping the shell stationary. A typical agitated mixer consists of a stationary shell (vertical or horizontal) with a single or twin shafts on which agitating devices are mounted. During mixing, particles are thrown randomly and the product is sheared or fluidized mechanically, depending on the tip speed of the paddles or plows. These mixers can handle a wide range of bulk solids from free-flowing to cohesive to pastes. While mixing is taking place, one can incorporate a liquid injection for further agglomeration and choppers or delumpers for breaking up the agglomeration, depending on the requirement.

In agitating mixers, the mixing is predominantly due to particles moving randomly from one point to the other, along with the bulk mass. So there is a combination of both shear and convection occurring within the mixer. Depending on the handling characteristic of powder, a certain degree of aeration will take place, and at higher tip speeds, the mass is capable of being fluidized. Once the material is aerated, the frictional effects including the interparticle forces are reduced, and sometimes eliminated, which enables the particles to move freely and randomly.

Paddle and Plow Mixers. The mixer typically has a single or double U-shaped trough with an impeller that consists of a single shaft or twin shafts mounted with plows/paddles at regular pitch in between, as shown in Figure 15-34. The plow helps to lift the solids creating chaotic motion causing shear in the powder mass that results in mixing. As shown in Figure 15-35, the motion of the powder in the mixer results in convective mixing whose intensity is proportional to the tip speed of the impeller.

At lower speeds, which is called *cascading*, the powder is carried by rotation and descends by rolling and/or sliding along the surfaces of the solids mass just as in tumbling mixers. At medium speeds, which is called *cataracting*, the powder is carried by the plow and drops either by sliding, rolling, or cascading. At higher speeds, which is called the *equilibrium regime*, the powder is mostly lifted by

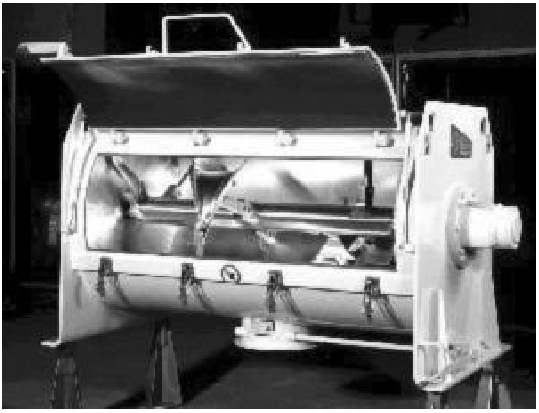


Figure 15-34 Plow mixer (Courtesy of Scott Equipment Company.)

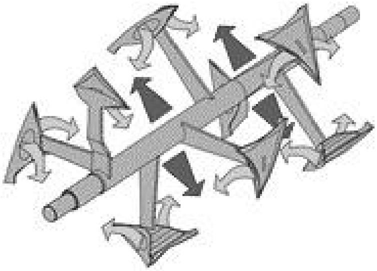


Figure 15-35 Plow mixer agitator and solids flow direction. (Courtesy of Kemutec.)

the plow and slides off at the end. In the equilibrium regime, there is hardly any chance for rolling, let alone shearing, and the desired mixing level will not be promoted. Hence, the right speed for operating the plow mixer depends on the physical properties of the product and the rotational speed of the mixer.

PRACTICAL INFORMATION FOR PLOW MIXERS

- *Typical size range for ingredients:* up to 5 mm, suitable for free flowing to slightly cohesive type of powders, can handle varying densities. Not suitable for very cohesive solids.
- *Mode of operation:* batch or continuous.
- *Choice of internal configuration:* single and double shafts with plows placed at regular intervals.
- *Suitability for special unit operations:* spray nozzle for agglomeration, high-speed choppers for breaking loose the agglomerates in the feed or mixture.
- *Mixing time/order:* up to 5 min with random mixing.
- *Particle degradation/attrition:* negligible when operated properly.

- *Type of industry:* food, ceramics, chemicals, building, plastics, pharmaceuticals.
- *Typical problems:* complete emptying is a problem.
- *Mechanical issues:* if steam cleaning with high-pressure water cleaning is adopted, mechanical seals must be selected to withstand the temperature and pressure limits. This requires regular maintenance. Seals will require routine inspection and maintenance.
- *Energy consumption:* up to 150 kW/m^3.

The paddle mixer (Figure 15-36) is similar to the plow type but the paddles mounted on the shaft differ in design from the plow mixer. Paddles can be oriented so as to impart lateral/back and axial mixing. The paddles are generally operated at higher speed than the plows. The speed has to be determined from running trials, and the right speed can result in good mixing. Higher speeds can cause segregation resulting in heavier product thrown near the wall and lighter product in the center of the mixer. Both the plow and paddle types can have double shafts with two horizontal impellers. The paddles or blades overlap those on the other shaft. In operation, the impellers counterrotate, fluidizing and mixing the material.

The paddle mixer can also be used for kneading and for mixing pastes since the kneading action allows phase changes from dry to paste when liquid is added. To evaluate the suitability of such mixers, it is recommended that test trials are conducted on the mixture to judge the duration and the quality of mixing. Paddle mixers are so versatile, they are also used as dryers and coolers for bulk solids.

PRACTICAL INFORMATION FOR PADDLE MIXERS

- *Typical size range for ingredients:* suitable for cohesive powders.
- *Mode of operation:* batch or continuous.

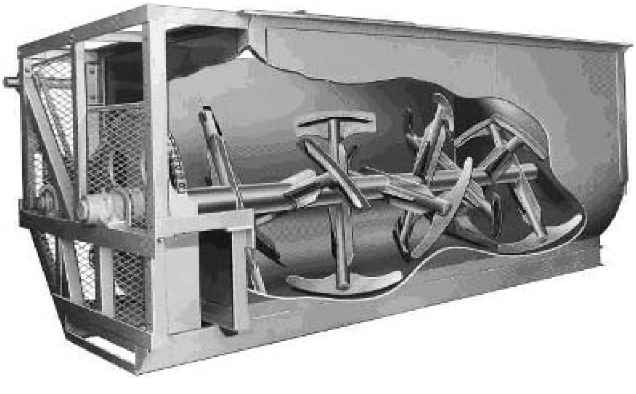


Figure 15-36 Single-shaft paddle mixer. (Courtesy of H.C. Davies Sons Manufacturing Co.)

- *Choice of internal configuration:* single and double shafts with paddles placed at regular intervals. These paddles can be oriented in such a way as to impart both axial and vertical mixing.
- *Suitability for special unit operations:* spray nozzle for agglomeration; high-speed choppers for breaking loose the agglomerates in the feed or mixture. Can be adopted for heating or cooling.
- *Mixing time/order:* up to 6 min with random mixing.
- *Particle degradation/attrition:* negligible. Higher paddle speeds can cause some attrition, but require quantification through trials.
- *Type of industry:* sewage treatment, dyes and pigments, animal feed, building, pharmaceuticals.
- *Typical problems:* clean up is easier. Complete emptying is not a problem.
- *Mechanical issues:* high speeds for the paddles calls for properly sized good-quality bearings and seals. Seals must be provided with air purge arrangements to insure that they stay dust free.
- *Energy consumption:* up to 150 kW/m^3.

Fluidizing Paddle Mixer (Forberg Mixer). A Forberg batch mixer [see also discussions by Forberg (1992) and Smith (1997)], shown in Figure 15-37, consists of paddles mounted on twin shafts in a twin trough. The ingredients to be mixed are fed from the top. The counterrotating paddles moves through the mixture throwing it in air, thus mechanically fluidizing the contents. Rapid fluidization is achieved. Discharge of the mixture take place through a large set of twin doors at the bottom of the mixer to minimize segregation. The peripheral speed of the paddle is about 1.5 m/s with gentle operation and very fast mixing of about 1 min (Forberg, 1992). Mixer volumes up to 50 m^3 are possible. Forberg mixers have been adapted to continuous mixing as well. Applications of Forberg

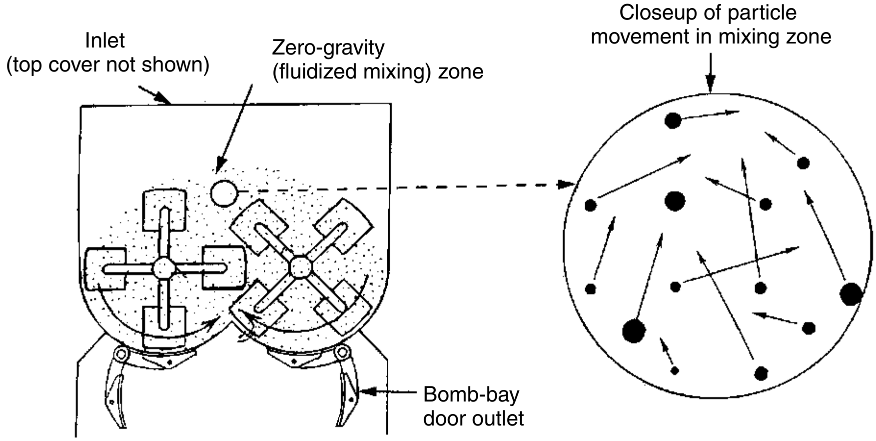


Figure 15-37 Forberg mixer.

mixers are in industries such as feed, food, pharmaceutical, chemical, building, and environmental.

MIXING TIME WITH A FORBERG MIXER. The time for mixing is dependent on the cohesion of the powders being mixed. As the cohesion increases, the mixing duration increases. However, the chances for the mixed solid to segregate while discharging are reduced due to solid cohesion. Normally, one requires a test at the supplier to verify the degree of mixing. The scale-up must be based on factors such as mixing duration, tip speed, solids filling point, and the discharge options after the process. Results related only to mixing time without such supporting information are useless (Muller, 1982). A comparison of the duration of a Forberg mixer to a generic plow mixer is shown in Figure 15-38. The results show that the paddle mixer is much more efficient then the plow mixer in terms of both mixing quality and duration. This is expected, since in the case of plow mixers, mixing is achieved primarily by diffusion and shear of the mass.

Ribbon Mixers

VERTICAL RIBBON MIXER. The vertical ribbon mixer [see also Pahl (1986), Steiss (1995), and Cavender (2000)] shown in Figure 15-39, is similar to a ribbon mixer turned upright. The mixer is designed to operate with 90% full of solids. During operation the ribbon rotates slowly. This action creates a shearing zone at the

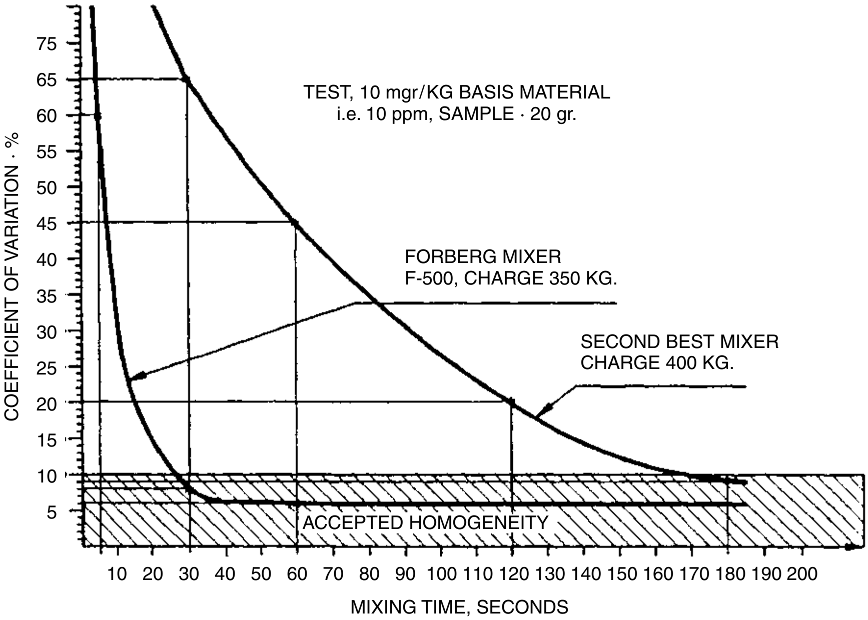


Figure 15-38 Forberg/twin shaft paddle mixer performance. (From Forberg, 1992.)

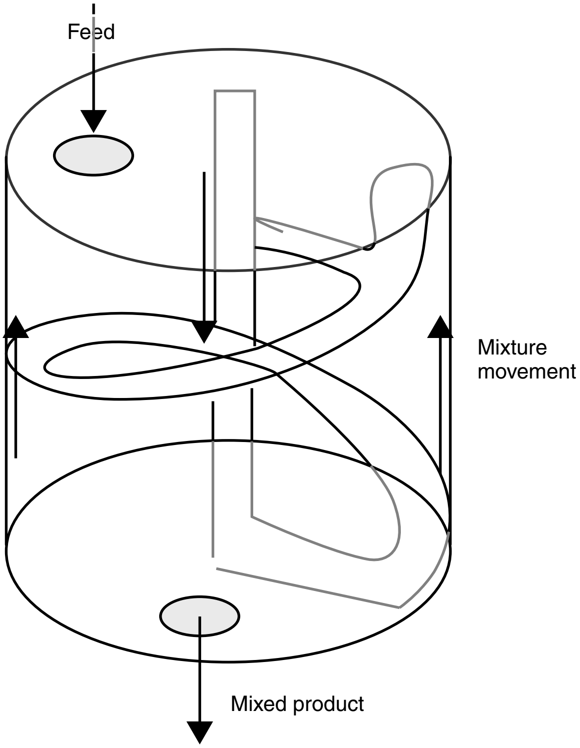


Figure 15-39 Vertical ribbon mixer.

wall, where the material moves in a helical fashion upward and flows down centrally. The mixer may be designed with a single or double shaft. The mixer with a single shaft has a capacity of up to 30 m^3 and can be operated under pressure or vacuum from 50 to 250°C. The mixer can handle friable products such as cereals, plastics, pigments, and pharmaceutical powders.

HORIZONTAL RIBBON MIXER. The ribbon (see Fuller, 1998) mixes the product in a trough by pushing it along the axis in both directions and displacing it by centrifugal force. Segregation may occur, resulting in separation into the bottom of the trough. The mixer is suitable for free flowing to cohesive products and is not suitable for sticky products. Emptying the entire contents may be difficult due to the small clearance between the trough and the ribbon. These mixers can be very large and the power required may be as high as 6 kW/m^3. A ribbon mixer, shown in Figure 15-40, has a double shaft, double helix, and top cover. The shaft has welded radial supports for the ribbons. The clearance between the ribbon's outer edge and the trough wall ranges from 3 to 6 mm. An inlet is located in the trough's cover at the top, and a discharge opening is either located in the center of the trough bottom or at either end. Figure 15-41 shows the direction of solids movement in such a mixer.

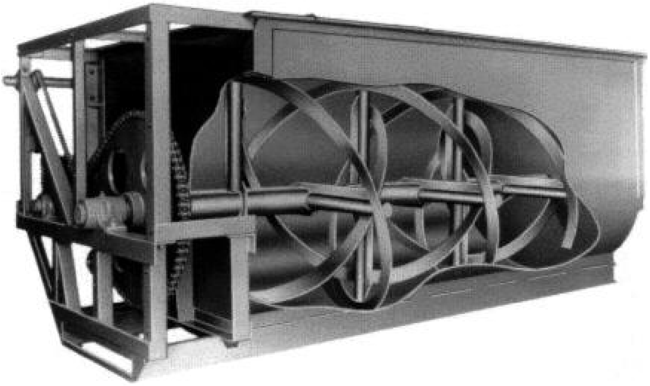


Figure 15-40 Horizontal ribbon mixer. (Courtesy of H.C. Davies Sons Manufacturing Co.)

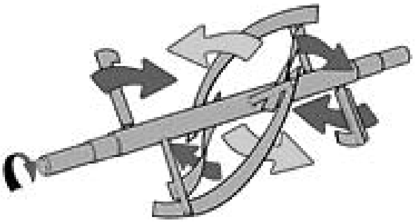


Figure 15-41 Direction of solids movement in ribbon mixer. (Courtesy of Kemutec.)

The mixture can be agglomerated by a liquid spray mounted above the ribbons. To reduce agglomerates, the mixer wall can be fitted with high-speed choppers. In operation, 40 to 85% of the mixer capacity is filled. The shaft rotates at relatively slow speed and the moving ribbons push the material back and forth. The inner ribbons move the solids toward the trough end, and the outer ribbons push the solids in the opposite direction, toward the center discharge valve.

For most powders, mixing can take 15 to 20 min. However, if the powders are fibrous, it could take much longer (up to 2 h) depending on the mixture quality requirements. During discharge, complete emptying can be a problem since a certain amount of product remains due to the clearance of the ribbon to the wall. This has to be cleaned out if ingredient change is required. These mixers can handle products ranging from free-flowing to cohesive powders and even slurries. Typically, they are used for adding a small amount of the ingredient to a larger bulk for general-purpose mixing.

PRACTICAL INFORMATION FOR RIBBON OR SHAFTLESS SCREW MIXERS

- *Typical size range for ingredients:* up to 5 mm, suitable for free to cohesive type of powders; can handle varying densities.
- *Mode of operation:* batch or continuous.

- *Choice of internal configuration:* single and double shafts with ribbons; single and up to three pitches designed either left- or right-handed to allow backmixing and conveying. Cut and folded screws are used for mixing of fine solids such as clay.
- *Suitability for special unit operations:* high-speed choppers for breaking loose the agglomerates in the feed or mixture. Can be adopted for heating or cooling.
- *Mixing time/order:* variable to 30 min, but can take as much as 2 h for fibrous products.
- *Particle degradation/attrition:* negligible. Particles can be broken at the clearance between the ribbon and the wall.
- *Type of industry:* widespread use. Food, chemicals, environmental, dyes and pigments, animal feed, wood and paper industry, pharmaceuticals.
- *Typical problems:* cleanup can be difficult. Emptying is not a problem.
- *Advantages:* emptying is not a problem.
- *Mechanical issues:* the ribbons must be designed to withstand the load equivalent to the volume of the mixer. The drive selected must be robust and allow for easier stop and start during mixing. Chunks, if caught at the clearance or in between the ribbons, can destroy the orientation of the ribbon. Care must be exercised to delump the agglomerates before feeding. Bearings are often gas purged. Material of construction depends on the product mixed.
- *Specific power consumption:* up to 12 kW/m^3.

Screw Mixers

VERTICAL ORBITING SCREW MIXERS. This type of mixer [see other discussions by Hixon and Ruschmann (1992), Pahl (1986), Steiss (1995), and Hosakawa Micron (1998)] consists of a hopper-shaped vessel and a screw feeder placed along the wall. The clearance varies from 3 to 6 mm, depending on the product. The screw orbits around the hopper but at the same time rotates in its own axis. This motion causes the mixture to lift and spread on a surface as shown in Figure 15-42. During this motion some shearing also occurs. It may not be suitable for products that are friable. Liquid can be introduced into the mixer through a suitable spray system. When two orbiting screws are used, one screw is normally shorter and is called a *satellite screw*. In some cases, two separate screws are used in two hopper vessels joined along the wall, as shown in Figure 15-42. Screw design can vary in flight design, pitch, and diameter. Recent designs incorporate varying diameter screws, which in fact reduces the ineffective area of mixing, thus increasing the residence time for particles.

PRACTICAL INFORMATION FOR VERTICAL OR ORBITING MIXERS

- *Typical size range for ingredients:* up to 500 μm, suitable for free to very cohesive type of powders; cannot handle varying densities.

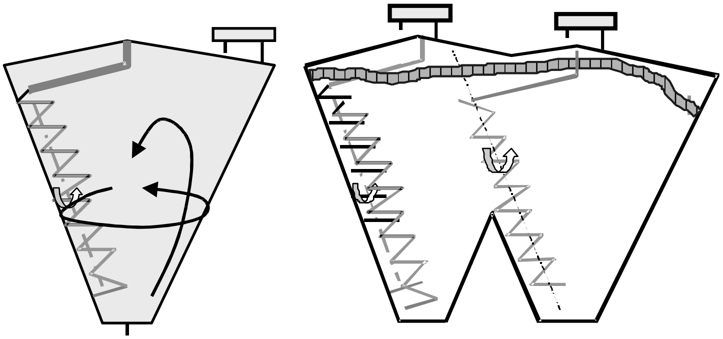


Figure 15-42 Vertical orbiting screw mixer.

- *Mode of operation:* batch only.
- *Choice of internal configuration:* screws can be driven from either above or below. A short satellite screw with normally larger diameter can be provided for better and faster mixing; screw designs vary from ribbon to screws with varying diameters to reduce the ineffective area of mixing. Spray nozzles can be mounted for liquid addition.
- *Suitability for special unit operations:* can be adopted for heating or cooling.
- *Mixing time/order:* at least 10 min.
- *Particle degradation/attrition:* friable products may degrade.
- *Type of industry:* food, chemicals, environmental, plastics, pharmaceuticals.
- *Advantages:* cleanup is not easy when sticky solids are handled. Emptying is easy.
- *Mechanical issues:* mechanical seals are used (Kent, 2002). These can be quite expensive and require special lubricants. There may be problems if this lubricant contaminates the product. Special seals are required in such cases.
- *Specific power consumption:* up to 80 kW/m^3.

Sigma-Blade and Z-Blade Mixers. These mixers [see also Pahl (1986), Fuller (1998), and Harnby (2000)] consist of twin troughs each fitted with a rotating agitator, and each one of these agitators is a heavy-duty Z-shaped blade, as shown in Figure 15-43. The product is introduced from the top of the mixer. The same access is used for cleaning purposes. The mixer can be tilted for emptying the products. A spray bar can be mounted above the blades. While in operation, the product is loaded up to 40 to 65% of the mixer's capacity. The blades can overlap and rotate at the same speed or at variable speeds. The mixing duration can be 10 to 30 min with good homogeneity of up to 99%. No delumper is required in this mixer. In Z-blades, two counterrotating Z-blades fold and shear the material quite severely. These are seldom used for dry solids, but are used for producing doughs and thick viscous pastes.

Comparison of Agitated Mixers. A classification of the rotating mixers or mixers with rotating components is made according to Froude number, Fr (Rumpf

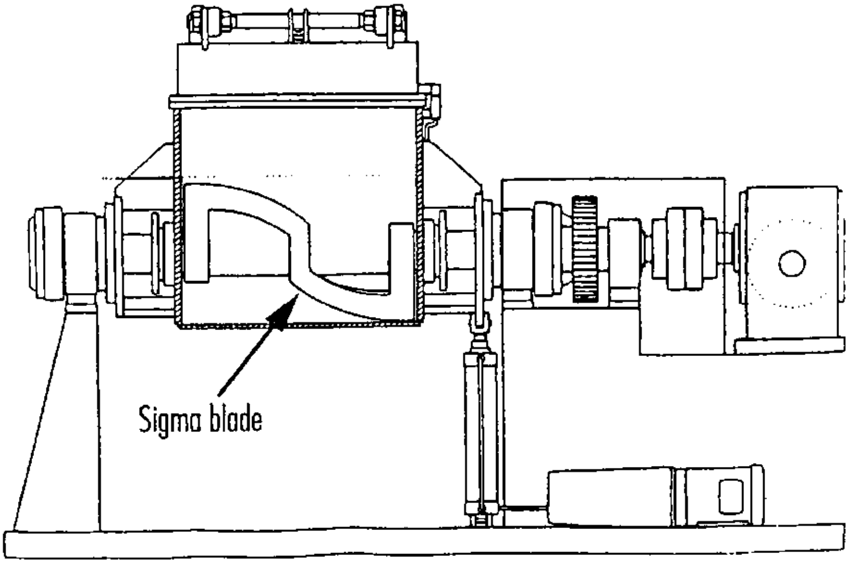


Figure 15-43 Sigma-blade mixer.

Table 15-2 Types of Equipment for Agitation of Solids by Rotating Inserts or Rotating Vessels

Type of Mixer	Motion by:	$\mathrm{Fr} = \frac{r\bar{\omega}^2}{g}$	Capacity (m^3)	Power (kW/m^3)
Tumbling type with rotating vessels; cylinders, drums, cubes, V and Y types, double cones	Free fall	<1	<2	<1–2
Double ribbon mixers, trough blenders, vertical screw types, orbiting type such as Nauta	Thrust	<1	<30	3–10
Centrifugal mixers with paddles plows	Thrust, centrifugal force	>1	<30	20
High intensity mixer Turbine and fluid mixers	Centrifugal force	$\gg 1$	<1.5	<500 20

Source: Rumpf and Muller (1962), Pahl (1986).

and Muller, 1962). Froude number is the ratio of the centrifugal force and acceleration due to gravity. Hence,

$$\mathrm{Fr} = \frac{r\bar{\omega}^2}{g} \tag{15-15}$$

where r is the radius of the mixer, ω is the angular velocity, and g is the acceleration due to gravity.

Tables 15-2 to 15-5 show different parameters of significance for agitator mixers. Table 15-2 shows different classes in batch mixers followed by the mode of operation and Froude number and respective capacity ranges and their power requirements. Note that only the high intensity mixers have $Fr \gg 1$, and centrifugal types with $Fr > 1$; otherwise, most of the mixers operate with $Fr < 1$. Sections to follow will treat each of the classes of mixers above.

This type of classification is one of the ways to characterize the mixer similar to liquid mixing; however, as Muller (Weinekotter and Gericke, 1999) points

Table 15-3 Comparison of Agitated Mixers

	Agitating			
Factor	Ribbon/Paddle	Plow	Fluidizing Paddle	Sigma-Blade
Material consistency	Powders/ granules	Powders/ granules	Powders/ granules	Pasty sticky gritty slurries up to 2×10^6 cP
Allowable fill level or batch size (% of total mixer capacity)	40–85	30–70	40–140[a]	40–65
Liquid addition configuration	Spray bar above ribbons	Spray nozzles at mixer top	Spray bar above paddles	Spray bar above blade
Delumping agitator configuration	High-speed chopper blades at sides	High-speed chopper blades at sides	Pin mills above paddles	None
Mixing cycle length (minutes)	15–20	<5	<1	10–30
Final moisture homogeneity (% of complete homogeneity)	90–95 or better	95–98 or better	98–99 or better	99 or better
Rotating or stationary vessel	Stationary	Stationary	Stationary	Stationary
Degree of particle shear	Some	High	Slight	Very high

[a] Percent fill more than 100% of the total capacity for another agitating batch mixer of equal volume.

Table 15-4 Comparison of Various Agitated Mixers

	Type of Mixer			
Property	Plow	Paddle	Ribbon	Shaftless Screw
Free-flowing powders, $50 < x < 500$ μm	Yes	Possible	Yes	Yes
Free-flowing granules, $200 < x < 5000$ μm	Yes	Possible	Yes	Yes
Cohesive powder	Possible	Yes	Yes	Yes
Energy transmitted to powder	Yes	Yes	No	Yes
Capacity (m^3/h)	1.8–1500	<1000	10–50	1–50
Size (m^3)	<40	<40	<50	<60
Filling ratio (%)	<70	<70	<60	<70
Specific power	10–150	10–150	3–12	8–12
Mixing time (min)	0.5–5	1–6	3–20	2–8
Froude number	1–9	<9	<1	<1.5
Cost/performance (U.S.\$/ft^3)	0.18	0.14	0.09	0.14

Source: Adapted from Ramponi et al. (2002).

Table 15-5 Typical Tip Speeds for Agitated Mixers

Type of Mixer	Tip Speed (m/s)
Ribbon	1.4
Turbine	3
Paddle	2–6
Twin shell tumbler with:	—
Pin-type intensifier	8.6
Liquid feed bar	17
Twin rotor	Up to 6.6
Single rotor	30–45
Mills of various types	12–100

Source: Adapted from (Weidenbaum 1973, Fayed and Otten 1984).

out, it has not yet been possible to characterize the solids mixer like the one for liquids. There is no relationship between a powder parameter that might be comparable to viscosity. Mixing of solids is by no means a straightforward task, and must never be underestimated. To appreciate and understand the mixing, one must understand the mechanisms influencing such an operation, and this is dealt with in the sections to follow.

Let us consider the case of a cohesive powder. During mixing of such a product, the ingredient may form lumps. These lumps may just circulate on their own without taking part in mixing. If the product is free flowing, one has to restrain the movement of particles, and this is contrary to the approach taken for mixing a cohesive product.

15-10.3.3 Gravity Silo Blenders. There are several instances in industry where the method of production or the nature of a process leads to variations in the quality of a particulate powder as a function of time. If these bulk solids are stored in silos before further processing or delivery to customers, these quality variations are propagated further in the process. In general, the contents of the silo are too large for eliminating these variations by the use of ordinary mixers, and homogenization has to be undertaken in situ.

Homogenization in silos can be undertaken using several techniques. The most common are fluidization, internal mechanical recirculation, and external recirculation with or without a hopper type of static mixing device. Since the variations in the quality of the stored powder occur as a function of time, the individual layers in the silo must be mixed with one another to obtain homogenization. Thus, one must design the hopper section of the silo with as large a hopper half-angle as possible to achieve the desired flow patterns. However, the flow patterns developed must be of mass flow to allow reliable bulk powder flow and to prevent segregation of the bulk powder upon discharge from the silo. Hence, the hopper half-angle should be designed on the mass flow/funnel flow limit to achieve the optimum blending efficiency of the silo.

The following classification along with Table 15-6 is helpful:

- Multitube blenders (refer to Figure 15-44)
  - Waeschle's gravity blender and combiflow blender
  - Phillips blender
  - Fuller blender
  - Mixing silo blender (Muller, 1982)
  - Zeppelin Centro blender

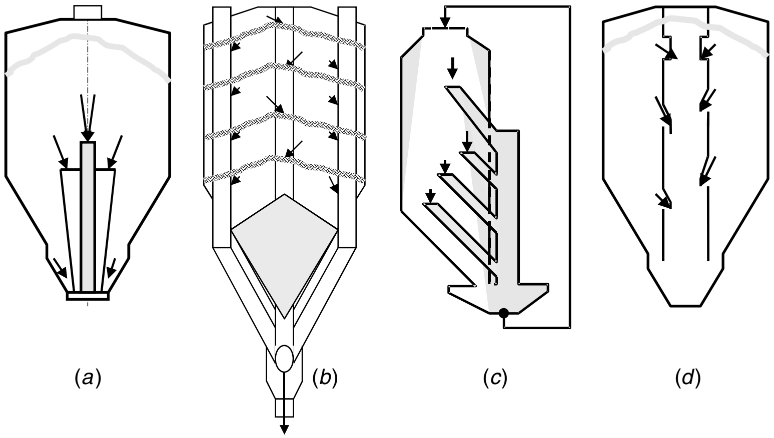


Figure 15-44 Multitube gravity blenders: (*a*) Waeschle's gravity blender and combiflow blender; (*b*) Phillips blender; (*c*) Mixing silo blender; (*d*) Zeppelin Centro blender.

Table 15-6 Survey of Blending Systems[a]

	Fluid Bed	Air Merge Type	Vertical Screw	Nauta Type	Zeppelin Type	Binsert Type	Waeschle Type	Phillips Type
Free-flowing powders $50\ \mu m < x < 500\ \mu m$	+	0	+	+	+	+	(+)	(+)
Free-flowing pellets $200\ \mu m < x < 5000\ \mu m$	0	0	0	0	+	+	+	+
Cohesive powders	−	+	+	+	(+)	+	−	−
Maximum vessel size (m^3)	1000−30 000	200	100	30−60	200	200	600	100
Energy required	Pneumatic	Pneumatic	Mechanical	Mechanical	Gravity	Gravity	Pneumatic	Gravity
Specific energy (kWh/t)	1−2	2−7	2−10	2−10	1−3	1−3	Ca2	<1

Source: Wilms (1992).

[a] +, Suitable; (+), Limited Suitably; −, Not Suitable; 0, Not Economical.

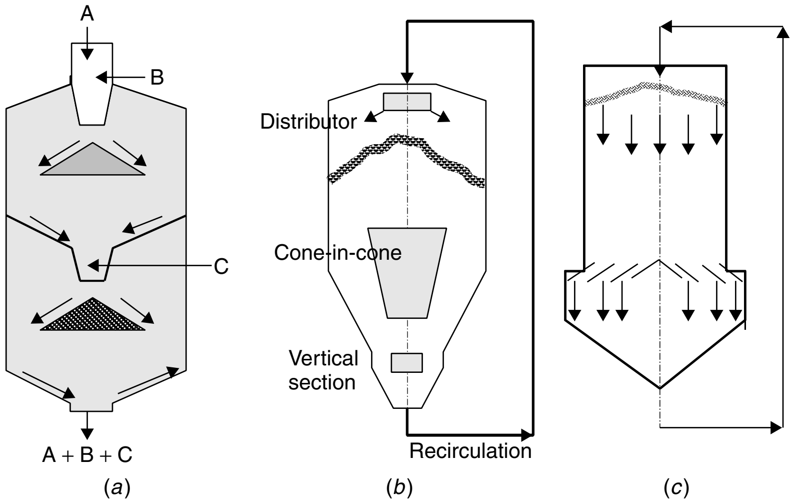


Figure 15-45 Blenders with inserts: (*a*) Roth blender; (*b*) Binsert blender; (*c*) Peschl blender. (As referred by Manjunath et al., 1992.)

- Insert-type blenders (refer to Figure 15-45)
  - Roth blender
  - Binsert blender
  - Peschl universal blender (Peschl, 1996)
  - Johanson blender with rings along the length
- Mechanical blender (refer to Figure 15-46)
  - Dual flow blender
  - Orbital screw-type blender (refer to Figure 15-42)

Various techniques have been used for homogenization of the silo contents. Figures 15-44*b*, and 15-45*a* show what are essentially multisilo arrangements. The Phillips blender, shown in Figure 15-44*b*, utilizes the principle of simultaneously drawing down powders from different levels in the silo and further promotes homogenization by providing two blending stages. The Roth blender in Figure 15-45*a* is a type of static mixer, where three product streams are mixed simultaneously.

Both principles are suitable for nonsegregating, uniformly sized particles whose physical or chemical characteristics may be marginally different. Figure 15-44*d* shows a Zeppelin Centro blender, and Figure 15-44*c* shows a mixing silo described by Muller (1982). In these types of blenders it is important to ensure that the discharge capacity of the central tube is larger than the combined inlet capacity of the ports along it and that the ports allow roughly similar amounts of powder to enter the central tube. Another blender manufactured by Zeppelin is a multipipe blender, which allows not merely powders from various levels to be mixed on

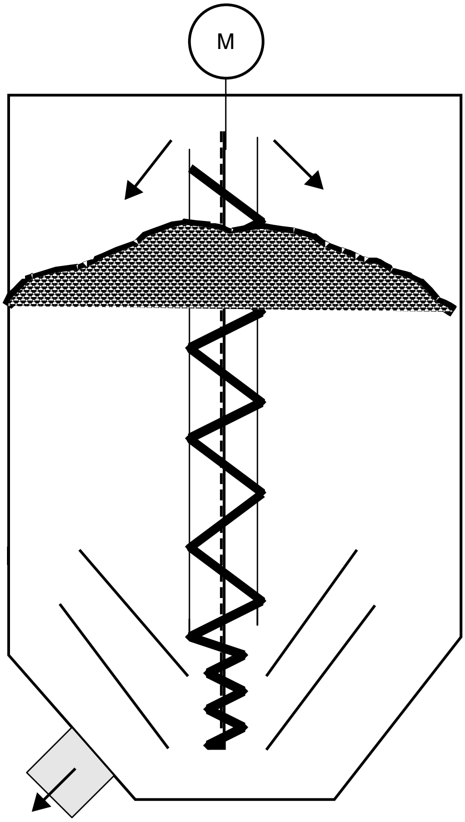


Figure 15-46 Mechanical blender.

discharge but also ensures that powders from various zones are also mixed. The disadvantages of these systems are that they are relatively costly to manufacture, and it is difficult to ensure that the various ports have similar capacities.

Figure 15-45 shows systems that use mechanical activation in addition to gravity. The Peschl universal blender (Figure 15-45*c*) also ensures mixing of the zones plus layers by creating a velocity profile across the silo diameter. This is achieved by applying differential levels of vibration to the concentric activating rings. It should be noted that care must be taken (Figure 15-46) to ensure that there is a differential rate of ingress to the screw from the various (concentric) zones in the silo to achieve effective blending.

It is clear that what one wishes to achieve is a mixing of the various zones plus the various layers in the silo. Doing this by gravity alone is considerably cheaper and more convenient than resorting to mechanical means. To achieve this blending objective, one must create a marked velocity gradient over the diameter of the silo while ensuring that the entire contents of the silo are in motion during discharge. As is also the case with most of the blenders described in the foregoing, it may be necessary to recirculate the contents of the silo several times to achieve an homogeneous mixture.

An effective method in which to create a large velocity differential of the type desired for a gravity blending is the Binsert hopper-in-hopper axisymmetric silo, shown in Figure 15-45*b*. Bulk material flows through the inner as well as through the annulus between the two hoppers. Placing the inner hopper at a predetermined position controls the velocity of the material. The design limits for this configuration are chosen near the limits for mass flow, to obtain a high velocity differential between the center and the outside unit. The critical point with reference to this type of blender is the aspect ratio, H/D, for the cylindrical section of the silo. Johanson calculated the required aspect ratio versus the hopper half-angle for a certain bulk powder, as shown in Figure 15-47. It is seen that the H/D ratio of 1.5 seems to be a limit for effective blending. It should be noted that as the hopper half-angle is increased to more than 35°, the aspect ratio necessary for effective blending is reduced drastically, to approximately 0.5.

Operation of Gravity Blenders. It is of considerable advantage to be able to predict the degree of blending that will be achieved in a given silo without the need to resort to experimental procedures on pilot scale test rigs. Consider a layer of markers, or marked particles, placed on top of a bulk powder, which is to be homogenized as shown in Figure 15-48. When the powder is discharged, some blending will take place. This degree of blending can be determined by plotting a graph of the percentage of markers in a sample against the volume of the powder removed and recirculated. During the first pass, a certain volume, V_1, must be discharged before any of the marked particles emerge from the silo. Johanson assumes an idealized triangular distribution as depicted in Figure 15-48 (Johanson, 1970). The volume, V_p, corresponds to the discharge of

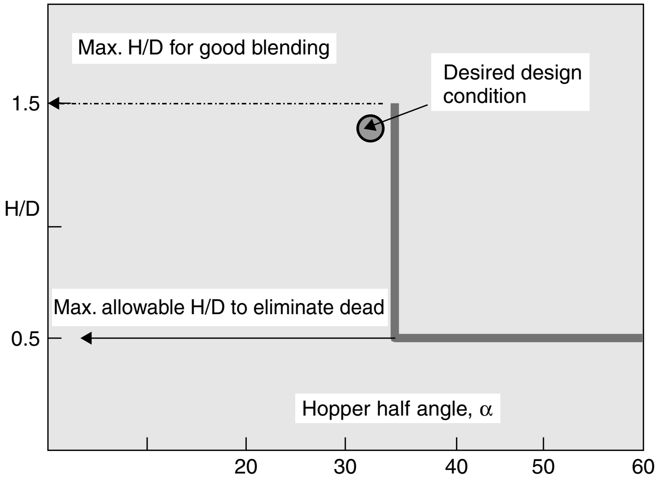


Figure 15-47 Optimal design of gravity blenders. (From Johanson, 1970.)

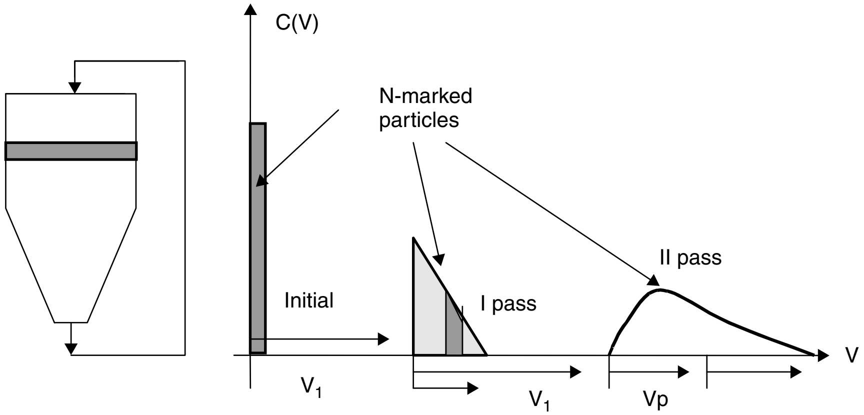


Figure 15-48 In-bin blending process representation. (From Johanson, 1970 and Roberts, 1990.)

the markers as the silo is emptied. During the second pass, the marked particles will be discharged over the volume $2V_p$ (Figure 15-48). After several passes the markers will be evenly distributed in the total volume. Effective blending can be obtained when $V_1/V_p < 1$. This implies that a portion of the markers must discharge as rapidly as possible after flow is initiated and that there must be a large time lag between the first marked particle to discharge and the last to discharge, for each cycle.

Silo–Feeder Interface (Manjunath et al., 1992). The well-designed blenders are often mismatched in terms of interfacing with discharge control equipments such as screw, belts, or vibratory feeders. Mismatching occurs due to either negligence or due to lack of information on the flow of solids. The result could be costly, since improperly interfaced feeders and silos often give rise to asymmetric flow patterns leading to variation in the quality of the products. Proper interfacing will help to overcome such problems. The following facts are useful:

- It is not enough to only design silos and vessels to operate in mass flow mode; it is equally vital to identify right discharge control equipment to ensure that the silo or reactor functions in the mode it is designed for.
- The small region between the silo and that of the discharge equipment, the region of interface, determines the flow pattern that is established in the blender.

Any negligence to proper interfacing results in variation of the product quality, which again calls for expensive retrofits. Such things are common in industry, causing loss in production time.

***15-10.3.4 Pneumatic Blenders*.** If powders exhibit expansion characteristics when aerated, they may be a good candidate for pneumatic blending. The expansion nature of powders can be determined by passing air through a permeable membrane or fluidizing media. The particles rise due to the drag force of the gas. Further increase in the air velocity, called the *superficial air velocity*, causes agitation in the bed, resulting in the formation of bubbles, causing mixing to take place. Such an operation (van den Bergh, 1994) is used for cement blending (10 000 m^3) and for blending of pellets (1000 m^3) in the petrochemical sector; however, fluidizing blenders consume very high energy, so industry also has considered gravity blenders. Refer to Table 15-7 for blender power consumption.

The amount of air to fluidize a system, the minimum fluidization velocity, is a function of the particle size, particle density, bulk density, and gas density. Bulk solids can be classified into four types, based on the density difference and mean particle size, as in Figure 15-49 (Geldart, 1973):

Table 15-7 Blender Power Consumption[a]

Blender Type	Power Consumption (kWh/t)
Air fluidized system	1–1.5
Gravity system, single inverted cone	0.25–0.5
Gravity system, multioutlet	0.1–0.13

[a] Power consumption Based on kWh/t of raw mill feed.

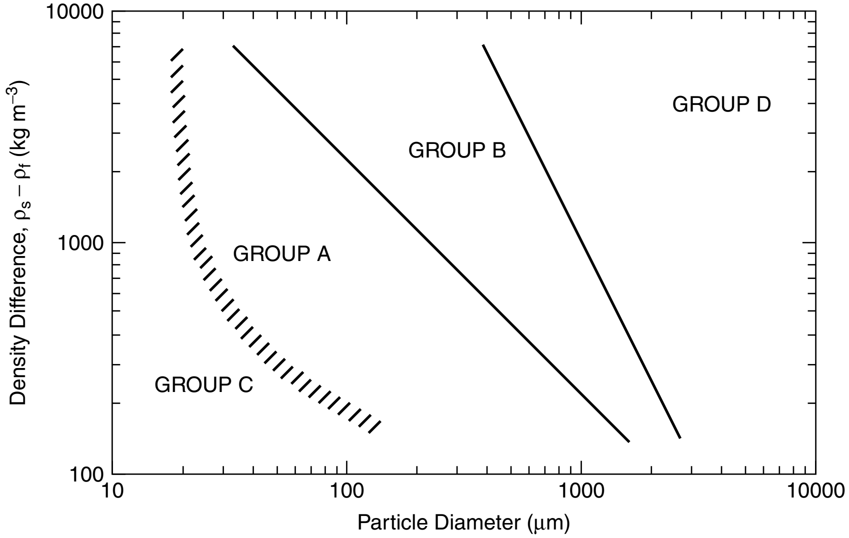


Figure 15-49 Geldart classification.

- *Class A:* aeratable, such as alumina; bubbling starts when the gas velocities are higher than the minimum fluidization velocity.
- *Class B:* bubbly; starts to bubble when the minimum fluidization velocity is reached.
- *Class C:* cohesive, such as cement; due to interparticle forces such as van der Waals and cohesion; cannot be mixed in fluidized beds.
- *Class D:* spoutable, such as plastic pellets; the required gas velocities are too high and spoutable beds can be an answer, not the fluidized beds.

Principle of Operation. The mixing action in the blender can be achieved by fluidizing the contents. If performed at velocities higher than the minimum fluidization velocity, the fluidization creates bubbles in the bed, except for C-type powders. Bubbles are necessary for mixing, as they drag solids in their wake, promoting mixing. Refer to Figure 15-50. A bubble consists a dome-shaped void plus particles in its lower region called the *wake*. As the bubble rises up to the top, solids are frequently exchanged between the wake and the drift. Finally, the bubble bursts forth from the surface of the bed and the powder is ejected from the bed. This process is largely responsible for blending or mixing of solids. The size and shape of a bubble depend on the particle/powder properties. To effectively express the degree of homogenization, it is essential to know the *bed turnover time*, and this is analogous to the number of silo volume recirculation in the case of gravity blenders. To evaluate the bed turnover time, modeling tools must be developed for better understanding.

The following three models in fluid–solid systems suggest how mixing takes place:

- Diffusion model
- Diffusion–convection model
- Convection model

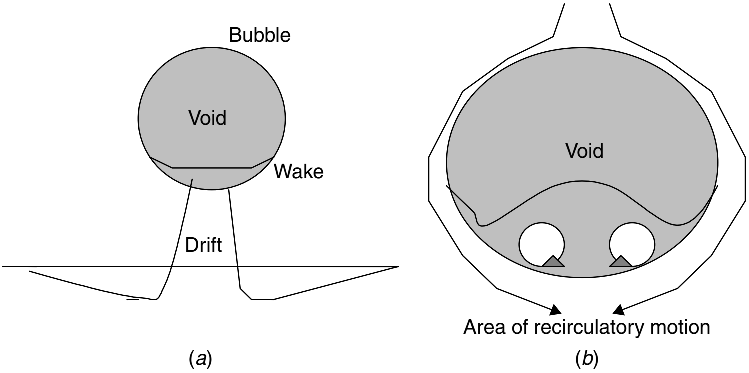


Figure 15-50 Bubble formation in a fluid bed blender: (*a*) bubble consists of a wake, drift, and void space; (*b*) circulation of solids around the bubble.

These models are also based on the Fickian equation (Lacey, 1954; Fan et al., 1990). The difficult part is the description of the diffusion coefficient for the diffusion model, which is a function of bubble size, its axial velocity, the particle density, particle size, the viscosity and density of the fluid, and the minimum fluidization velocity. When describing these models, experimentation is required for verification of the above parameters. Axial dispersion takes place when the bubbles are rising in the bed, and horizontal dispersion when the bubbles burst. However, there exists evidence that horizontal dispersion also occurs as the bubbles form and move up the bed due to continuous displacement of the mass around the bubble.

Comparison with Mechanical Blenders or Homogenizers. Mechanical blenders normally have recirculation systems either within the blender or placed outside it. There are several types, but they have in common steep hopper half-angles with respect to vertical as compared to fluid bed blenders. This is required to allow all the mixed material to flow out of the blender. The silo geometry has to ensure mass flow conditions, which accounts for even withdraw of products, but at the same time, the angles have to be somewhat shallower to allow shear between the flowing layers of products to cause blending. If the outer hoppers are shallower, then normally, blenders are equipped with inserts, such as hopper-in-hopper types. These inserts can work in both axisymmetric and plane flow blenders, although the former types are more common.

Types of Pneumatic Blenders. The following types are available:

- Air mix blender
- Air merge blender
- Modified air merge blender
- Pneumatic blender (Krambrock, 1976)
- Entire hopper bottom fluidized

There are several configurations of mixers available. Figure 15-51*a* is called the *Air mix*. To achieve mixing, the system employs a blower or compressor to generate air flow, a control valve to vary air velocity, and downstream of the blender, gas cleaning systems are required to capture the fines. The Air mix can operate in a closed cycle, so that any gas can be employed to fluidize the contents. The gas is introduced through the mixing head as shown in the figure. It is claimed that such a design produces a swirling turbulent action within the blender in a matter of 15 to 30 s, and a similar period is required for the particles to resettle.

On the other hand, the *Air merge* blender employs a hopper divided into several segments, and each segment can be fluidized (Figure 15-51*b*). The fluidization of these quadrants or segments can be controlled. A modified version of the Air merge blender is the column blender shown in Figure 15-51*c*, which consists of a central draft tube through which powder can flow during mixing, thereby

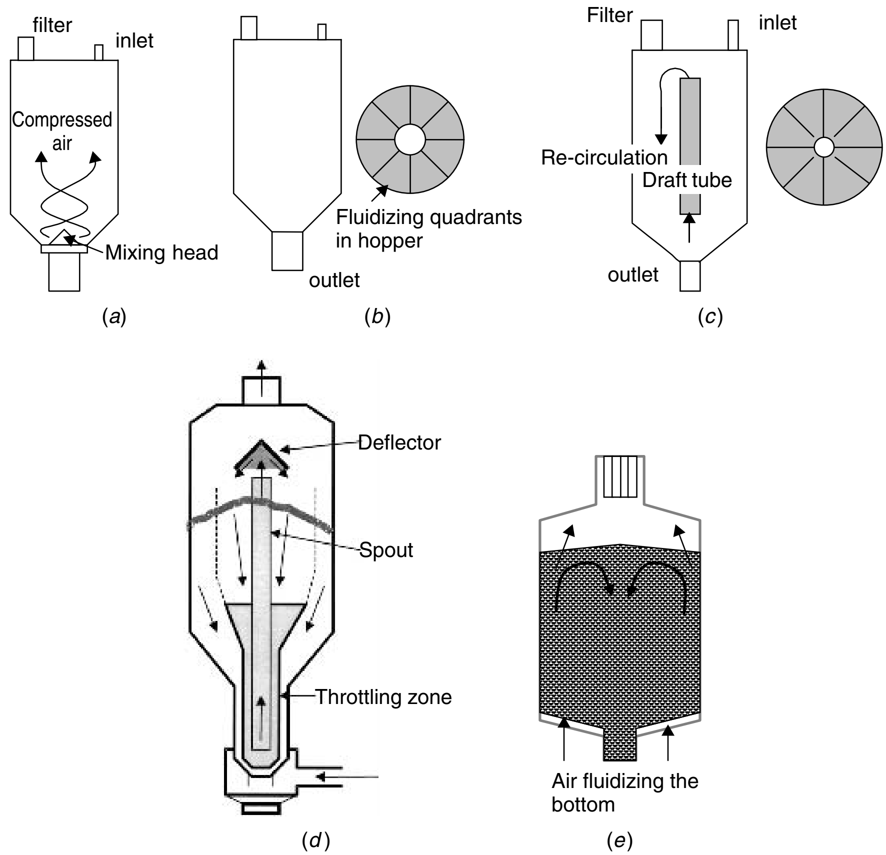


Figure 15-51 Pneumatic and fluid bed blenders: (*a*) Air mix; (*b*) Air merge; (*c*) modified air merge; (*d*) pneumatic blender (Krambrock, 1976); (*e*) entire hopper bottom fluidized.

increasing effectiveness during the process. The blending area is located directly in the center of the silo. It is claimed that the powder flows up the draft tube, overflows at the top, and flows down the outside of the tube and into the bottom of the tube for recirculation. Certain blenders have hopper parts completely fluidized without sectoring the bottom, unlike the two types above. Fluidization of the mixture is important and causes the development of bubbles for achieving mixing. While discharging from such hoppers, a velocity below the minimum fluidization value, called *deaeration velocity*, must be used for better emptying of the contents, which are well mixed, else segregation of the mixture will result. Krambrock (Figure 15-51*d*) focused on the petrochemical industry to blend plastic granules in large quantities, particularly for obtaining uniform product quality, such as melt index, color values, and the number of additives during long production periods. Table 15-8 contains the design and operating data of some mixer sizes suitable for plastic granules. It is important to note that the mechanical aspects of fluidizing

Table 15-8 Design Data for Homogenizers

Volume (m^3)	Mixing Time, for 3 Recirculations (h)	Total Air (SCM/h)	Diameter of Blender, D (m)	Height of Mixer, H (m)	ΔP without Filter Piping (mbar)
1	0.25	1100	1	2.9	32
	0.5	550			
	1.0	275			
10	0.95	5400	2	6.3	45
	1	2700			
	2	1350			
100	2	14 000	3	19.4	90
	4	7000			
	8	3500			
200	4	14 000	3.5	28.2	120
	8	7000			
	16	3500			

Source: Krambrock (1976).

silos must be carefully designed because of the possibility of development of hydrostatic pressures in the silo.

The pneumatic blender (Figure 15-51*d*) consists of a mixing silo, a central conveying tube, and a cone at the top for spreading the mixture. During filling the outlet is closed and the slide valve prevents entry of product into the fan. The cone prevents product from falling back into the central tube. Once the blender is filled, air is introduced through the central tube; the product is carried with it and spreads at the top as it hits the cone. This motion is carried out for a certain duration, for mixing. In this way, the inner and the outer product layers are displaced relative to one another in the axial direction, to achieve good mixing. The capacity or throughput is determined from the gap between the tube and the annular region; as the gap increases, so does the capacity. This also increases the pressure drop.

15-10.3.5 High Intensity Mixers. An impaction mixer (the Henschel mixer) is shown in Figure 15-52. Granules repeatedly break as they form and re-form. The shape of these mixers lends itself to easy cleanup and maintenance. The impaction mixer resembles a typical kitchen food processor. The blades rotate at speeds within the range 2000 to 3000 rpm, so these mixers require significant energy compared to other types with similar capacities (Harnby, 1992). The impaction mixer is used as a mixer–granulator.

15-10.3.6 High-Shear Mixers. Harnby states; “These are the alchemist’s mortar and pestle and the miller’s milestone for grinding of grain” (Harnby, 2000). As powder is pressed between two pressurized rolls (Figure 15-53*a*), any agglomerates will be pulverized. These mixers are commonly preceded by a

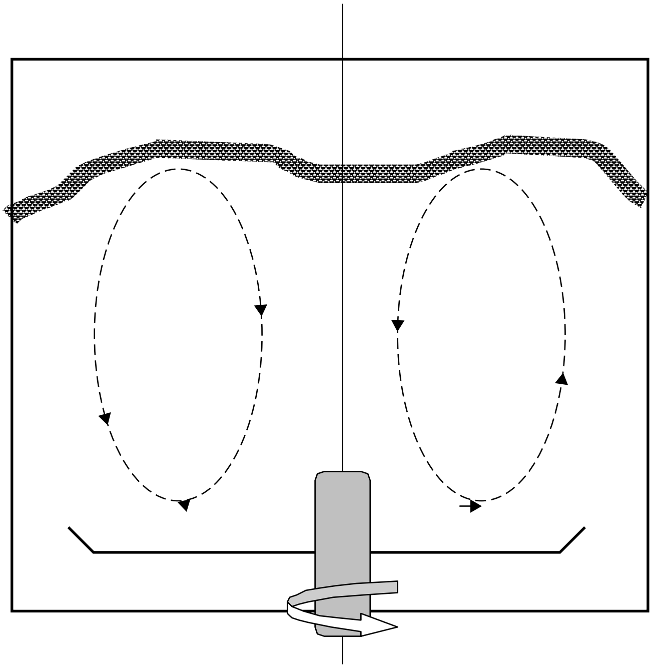
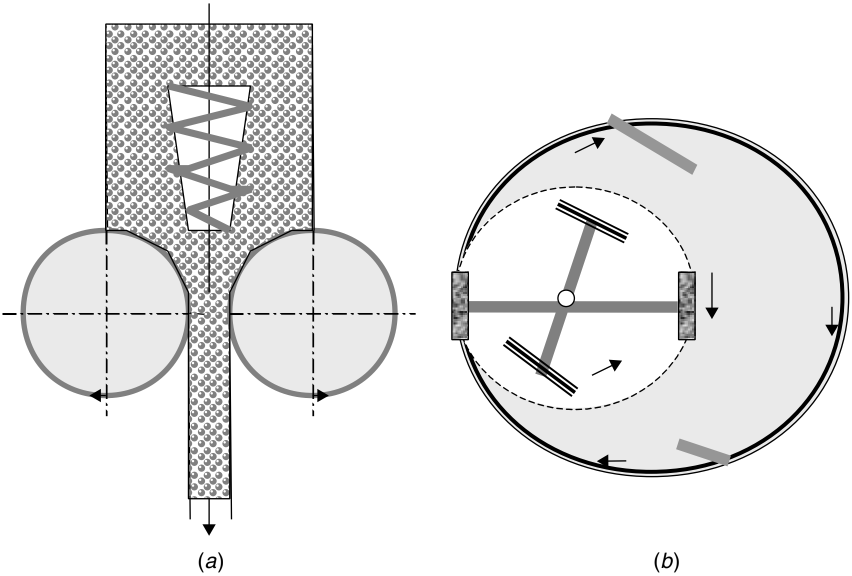


Figure 15-52 Impaction mixer (Henschel mixer).



(*a*) (*b*)

Figure 15-53 (*a*) High-shear mixer (Harnby et al., 1992) and (*b*) Muller mixer (Weidenbaum, 1973; Fayed and Otten, 1984.)

convective tumbler mixer to provide a reasonable quality before the product is conditioned. The muller mixer is intended for finely ground powders and is available in both batch and continuous modes of operation. The turret and the pan can be designed to rotate in opposite directions. Some designs have a stationary pan with only the turret rotating. As shown in Figure 15-53*b*, the rollers grind the material into a very finely divided and well-mixed consistency by high shearing, and at the same time by folding and turning the mixture over in each turn, resulting in intimate mixing of ingredients. It is not easy to clean or to empty these mixers.

15-11 CONTINUOUS MIXING OF SOLIDS

Continuous mixing is the preferred option for processes where throughput is high, space is a constraint, storage of intermediates must be avoided, or the material has a tendency to segregate. A continuous mixer can be a low-cost and reliable option if the operating variables are appropriately controlled. Selection of batch versus continuous mixing has been discussed in Section 15-9.

Continuous mixing is achieved by homogenization of incoming component streams at a fixed rate and ratio. The time required to obtain a homogeneous mix determines the size of a continuous mixer. If the streams are being fed side by side in a mixer, it is the radial mixing that dictates the mixer efficiency. Radial mixing can be achieved by agitated internals (paddles or plows), by tumbling the shell (zigzag mixer) or by static internals in a static mixer. The size of a continuous mixer based on radial mixing alone would be extremely compact. However, it is very challenging to feed fine particulate consistently without fluctuations. This problem is magnified when a mixture contains multiple components.

Axial mixing can dampen the effect of feed stream fluctuations on the composition of the mixture at the outlet. The greater the axial mixing, the lower is the influence of fluctuation or inconsistencies of feed stream. Danckwerts (1953) proposed a variance reduction ratio (VRR) to quantify this characteristic.

$$\text{VRR} = \frac{\sigma^2_{\text{input}}}{\sigma^2_{\text{output}}} \tag{15-16}$$

A good mixer will have a high VRR when σ^2_{output} is low, even when σ^2_{input} is high. The ratio above is a metric for the performance characteristics of a continuous mixer. For a given mixer, this ratio also depends on the operating parameters and the nature of input variation. To demonstrate this relationship, Weinekotter and Gericke (1999) carried out detailed investigations using the setup shown in Figure 15-54. Input disturbances of different time periods were imposed on the mixer. The output concentration was monitored using an optical probe. The results are summarized in Figure 15-55.

When the time period of input fluctuation (120 s) is greater than the residence time (44 s), the mixer is incapable of dampening out the fluctuations. The input

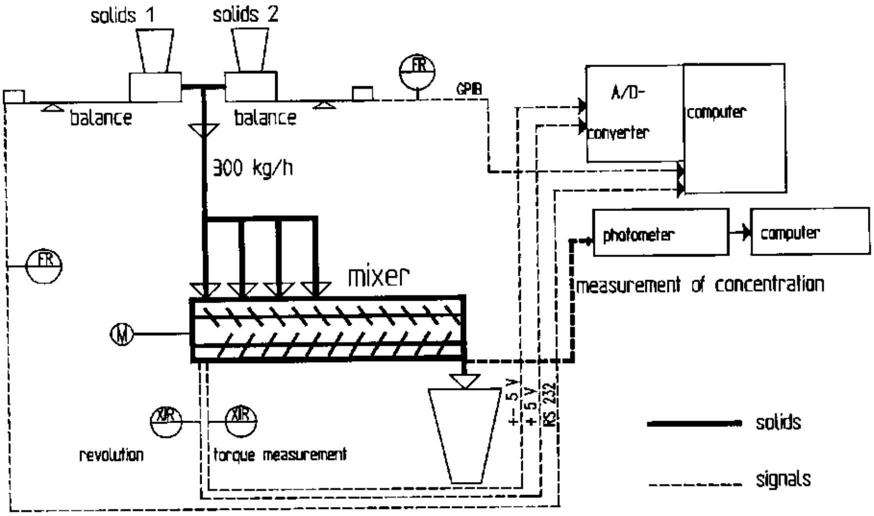


Figure 15-54 Experimental test set for continuous mixers. (From Weinekotter and Gericke, 1999.)

variability is propagated to output concentration and results in a VRR of 1. On the other hand, when the time period of the input fluctuations is 30 s, a significant dampening is observed and the resulting VRR value is 82. In general, higher-frequency fluctuations in the feed stream are easier to dampen out than low-frequency fluctuations.

Therefore, one must consider the relationship between average residence time and possible cycle time of feeder fluctuations while specifying a continuous mixing system. The average residence time in a mixer is given by

$$\text{average residence time} = \frac{\text{retention volume in mixer}}{\text{mass flow rate/bulk density}} \tag{15-17}$$

The retention volume refers to the volume of solids in the mixer at any instant.

Given the stochastic nature of the motion of particles in a mixer, it is common to observe a distribution of residence time. This is a direct result of axial mixing or dispersion. Axial dispersion/mixing will help dampen the feed fluctuations; however, it also broadens the residence time distribution. A broad residence time distribution implies a variable time history for particles passing through the mixer. If the mixer is used as a dryer, granulator, coater, or reactor, broad residence time distribution is not desirable. A balance between the process requirements (allowable residence time distribution) and process capability (feeder characteristics and mixer residence time) must be reached to design a robust continuous mixing system.

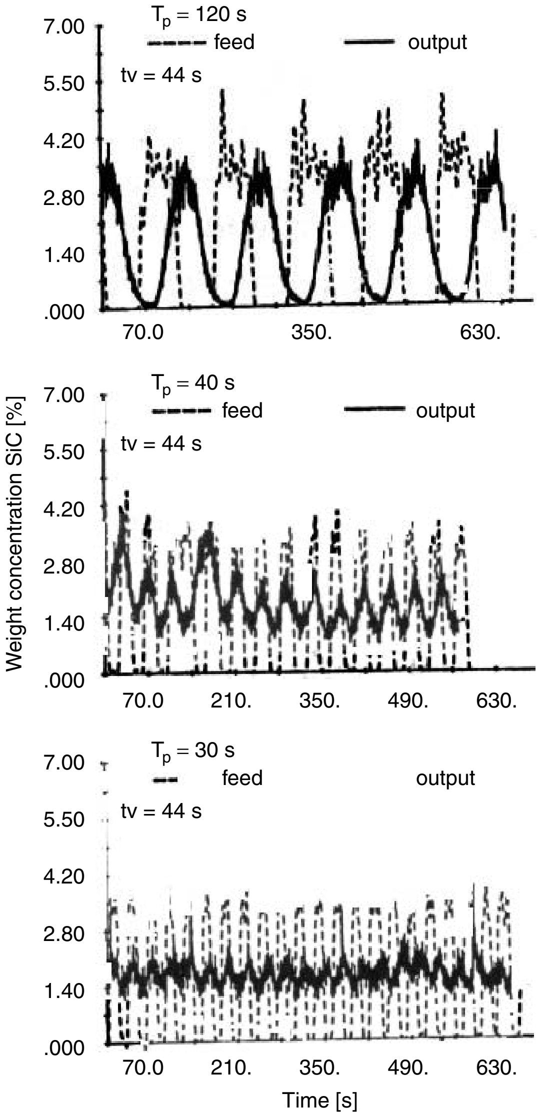


Figure 15-55 Relationship between residence time and feed fluctuation reduction. (From Weinekotter and Gericke, 1999.)

15-11.1 Types of Continuous Mixers

15-11.1.1 Mixing by Simultaneous Feed. Mixing using two or more feeders represents a continuous mixing process. Metering each ingredient precisely and bringing the streams together blends the components. For free-flowing solids, a static mixer can be used. There is little chance for backmixing or axial mixing in such units. Static mixers must always be starve-fed to achieve proper randomization of feed streams.

15-11.1.2 *Agitated Mixers as Continuous Mixers*. Ribbon and paddle mixers can be run in continuous mode. The material is fed at one end of the mixer while a weir on the opposite end provides retention control. The average residence time and residence time distribution depend on the mixer speed, geometry of agitators, and the rpm. These mixers are typically run 30 to 50% full during normal operation. Typical residence times are in the range 0.5 to 5 min. Some agitated mixers, such as the Nauta (vertical screw) and plow types, are not suitable for operating in continuous mode.

15-11.1.3 *Tumbling Mixers as Continuous Mixers*. Rotating drum or zigzag mixers are commonly used as continuous mixers. These are designed with a high aspect ratio and operated in horizontal mode. Continuous drums are inclined slightly for natural movement of material along the length. These are particularly suitable for coating applications, where low intensity mixing and long residence time are required.

15-12 SCALE-UP AND TESTING OF MIXERS

In this section we outline the commonly accepted scale-up criteria used in industry for agitated mixers and silo blenders. Scale-up of tumbling mixers was addressed in Section 15-5. The complexity of interaction between physical properties of solids, mixer configuration and velocity, and stress profiles within a mixer makes it difficult to formulate generalized scale-up criteria. Various experimental investigations into scale-up, however, do provide useful guidance for scale-up. No concurrence on acceptable approach has been reached, and various manufacturers tend to follow their experience.

A general axiom for scale-up is: "Commit your blunders on a small scale, and make your profits on a large scale" (Zlokarnik, 1984). Although it is easy to conduct experiments on a small scale, one must also consider the following questions:

- How small can the pilot mixer be?
- What geometric, dynamic, and kinematic parameter ratios need to be kept constant to ensure the validity of data on a large scale?
- Are the material properties (e.g., particle size distribution, temperature, and moisture) comparable to those in a large scale process?
- Are there other processes (heating, cooling, granulation) that require scale-up along with the mixing process?

15-12.1 Principle of Similarity

Similarity principles are applied routinely for scaling up of industrial liquid–liquid mixers. Wang and Fan (1978) first proposed using geometric, kinematic, and dynamic similarity for scale-up of tumbling mixers. *Geometric similarity* involves keeping the ratio of linear dimensions of pilot and full scale mixers constant. *Kinematic similarity* requires the velocity ratio between corresponding points in the two (pilot and full scale) systems to be constant while maintaining geometric similarity. *Dynamic similarity* dictates that the ratio of forces at corresponding points in the two systems be constant. Sometimes, nondimensional force ratios (e.g., Froude number, Reynolds number) are used. When the governing equations are not known, such parameters can be derived from dimensional analysis.

An industrial practitioner is interested in the following questions:

- What are the power consumption and mixing time in a full scale mixer for a given quality of mixing?
- How do the mixing quality and power consumption change with mixer rpm?

15-12.2 Scale-up of Agitated Centrifugal Mixers

As mentioned earlier, it is nearly impossible to formulate generalized scale-up equations for solids mixers. However, extensive experimental investigations conducted by Muller (1982), Scheuber et al. (1980), and Merz and Holzmuller (1981) have resulted in the following usable criteria. As shown in Figure 15-56, two regions are demarcated at a Froude number of 3. The improvement in mixing coefficient for a given mixer at Fr > 3 is dramatic.

The coefficient of mixing, M, proposed by Muller, is a parameter used in his semiempirical one dimensional model of horizontal mixers. The mixing coefficient determines how quickly concentration equalization will occur in the mixer. A large mixing coefficient will result in a short mixing time for a given quality of mix. The mixing coefficient is assumed to remain constant at all points in the mixer for the duration of the mix. It should be noted that M depends on the type of mixer, geometry of the internals, and the operating conditions. It does not depend on the properties of the mixture components (e.g., size or density).

$$\frac{M}{D^2 n} = \text{constant} \quad (\text{Fr} < 3) \tag{15-18}$$

$$\frac{M}{D^2 n} \approx \text{Fr}^2 \quad (\text{Fr} > 3) \tag{15-19}$$

where M is the mixing coefficient, D the diameter of the mixer (m), n the mixer rpm, and Fr the Froude number, defined as

$$\text{Fr} = \frac{v^2}{gR} = \frac{R\omega^2}{g} \tag{15-20}$$

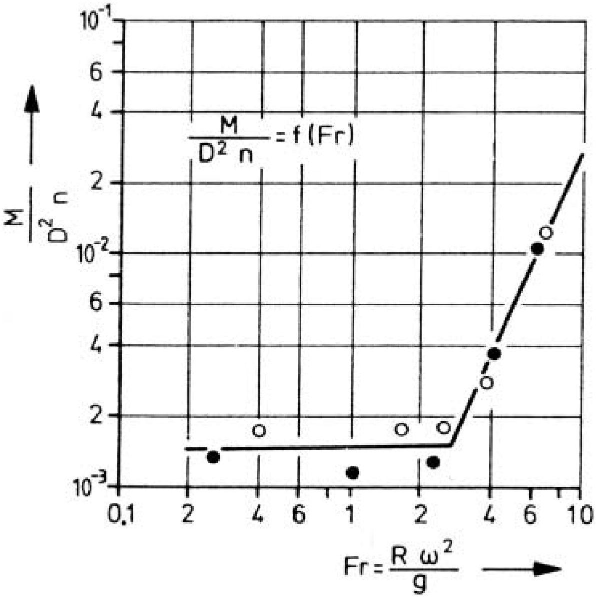


Figure 15-56 Relationship between dimensionless mixing coefficient and Froude number. (From Muller, 1982.)

where v is the peripheral velocity of mixing element (plow, paddle) (m/s), R the mixer radius (= D/2) (m), and ω the angular velocity of the agitators (rad/s):

$$n = \frac{30\omega}{\pi} = \frac{30}{\pi}\left(\frac{v}{R}\right) \tag{15-21}$$

Two common approaches are used for scaling these mixers:

1. Keep the peripheral speed constant between the pilot mixer and the full scale mixer.
2. Keep the Froude number constant between the pilot mixer and the full scale mixer.

Note that each of these approaches is used by mixer equipment manufacturers, and this suggests that more research and development are required to increase our understanding of solids mixing processes.

- *Criterion 1: Keep* v *constant:*

$$\frac{n_{\text{pilot}}}{n_{\text{full scale}}} = \frac{R_{\text{full scale}}}{R_{\text{pilot}}} \tag{15-22}$$

assuming geometric similarity and the same quality of mixing.

- *Criterion 2: Keep Froude numbers constant:*

$$\frac{n_{\text{pilot}}}{n_{\text{full scale}}} = \sqrt{\frac{R_{\text{full scale}}}{R_{\text{pilot}}}} \tag{15-23}$$

assuming geometric similarity and the same quality of mixing.

It is a common practice to use a Froude number of 7 for mixing nonfriable materials. For friable materials, the effect of agitator impact on breakage must be evaluated. Attrition is nonlinear with impact velocity, whereas it is linear with mixing time. Therefore, an optimum can be found through experimentation.

15-12.2.1 *Mix Time*. Rumpf and Muller (1962) have shown experimentally that the mixing coefficient can also be related to mixer length (L) if the mixer diameter (D) is kept constant:

$$\frac{Mt}{L^2} = \text{constant} \tag{15-24}$$

where M is the mixing coefficient, t the mixing time (s), and L the mixer length (m).

For Froude numbers below 3 and for geometrically similar mixers operating at the same peripheral speed of agitator, the mixing time increases linearly with the mixer diameter,

$$t \approx \left(\frac{L}{D}\right)^2 \frac{D}{v} \tag{15-25}$$

where D is the mixer diameter.

At higher Froude numbers (>3), the mixing time is linear with mixer volume (not the diameter). The effect of agitator speed is significant in this range.

$$t \approx \left(\frac{L}{D}\right)^2 \frac{D^3}{v^5} \tag{15-26}$$

15-12.2.2 *Power Consumption*. The relationship between power consumption and Froude number for agitated centrifugal mixers is shown in Figure 15-57. The power consumption is expressed in a nondimensional form as the Newton number (Ne):

$$Ne = \frac{P}{\rho_s(1-\varepsilon)D^5 n^3 \left(\frac{L}{D}\right)} \tag{15-27}$$

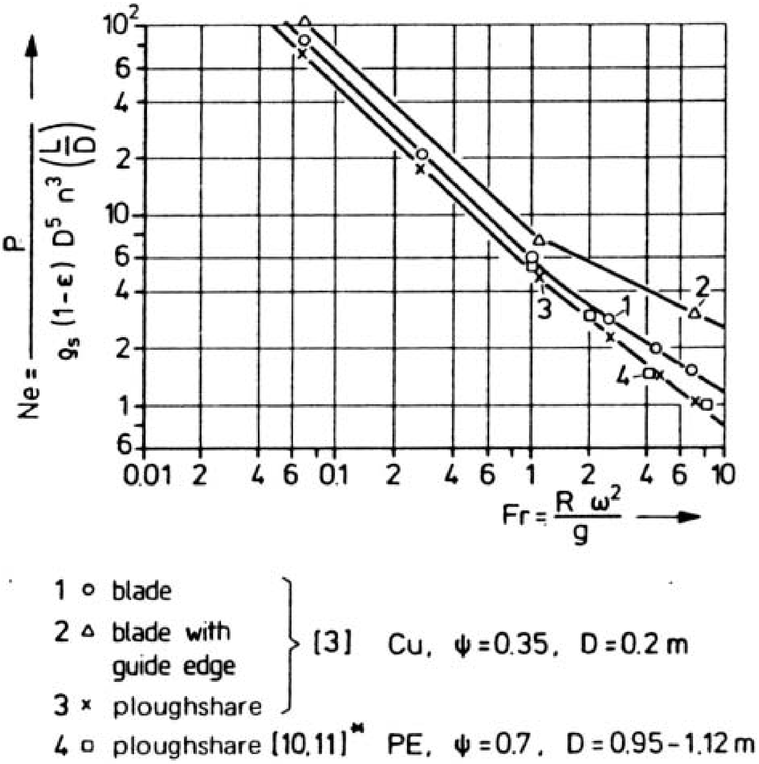


Figure 15-57 Relationship between power consumption and Froude number for centrifugal agitated mixers. (From Muller, 1982.)

For Fr < 1, where the acceleration forces are relatively small, the material is not fluidized and under plastic shear, the following relationship holds:

$$\mathrm{Ne} \sim \mathrm{Fr}^{-1} \qquad (15\text{-}28)$$

At higher Froude numbers, the configuration of paddles/agitators will have a significant influence on the shape of the curve (Figure 15-57). The surface roughness, shape, and size of the particles also have a significant influence on this curve.

In practice, the driver motor and shaft must be designed such that the mixer can be started with a normal load of material. The peak torque and power associated with the startup conditions far exceeds the operating conditions.

15-12.3 Scale-up of Ribbon Mixers

The literature on mixing in ribbon mixers is not extensive. The following rules can be extracted from current literature:

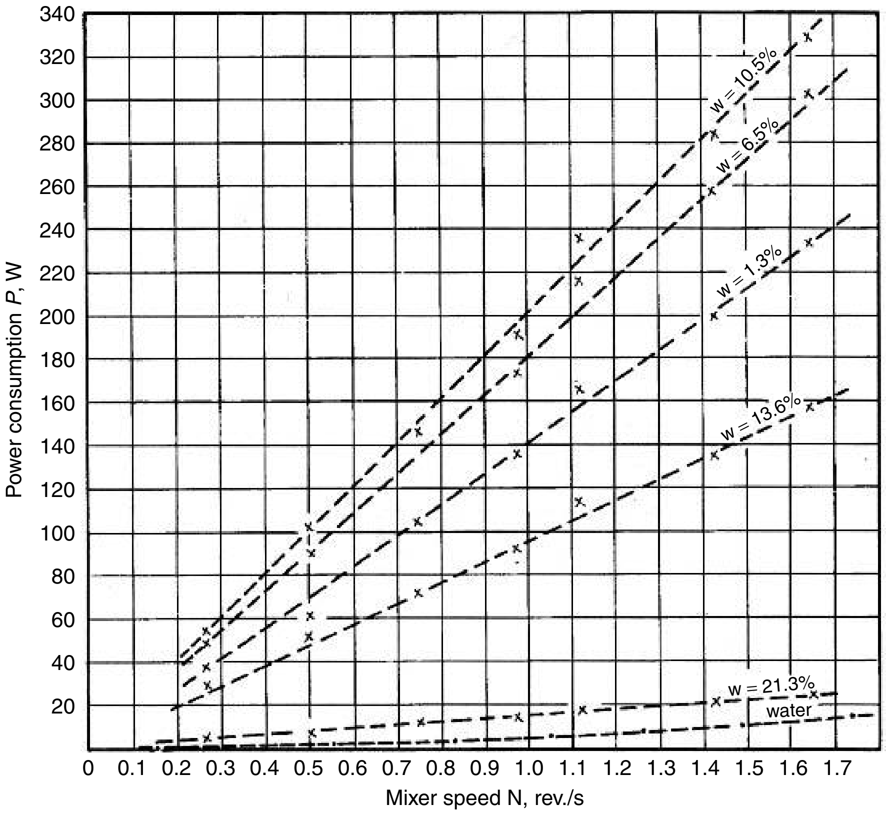


Figure 15-58 Variation of power consumption with rpm. (From Masiuk, 1987.)

- Power consumption in a ribbon mixer increases linearly with rpm at a given level of fill in the mixer. The rate of increase in power consumption with rpm is greater at higher fill levels (see Figure 15-58).
- The power consumption depends largely on the pitch and diameter of the mixing ribbon rather than its width.

15-12.4 Scale-up of Conical Screw Mixers (Nauta Mixers)

Entrop (1978) conducted a wide range of experiments in conical screw mixers. The results can be summarized as follows:

15-12.4.1 Power Consumption

$$\frac{P}{n_s \rho_s (1-\varepsilon) d_s^4 g} = k_1 \frac{n_s}{n_a} \left(\frac{1}{d_s}\right)^{1.7} \qquad (15\text{-}29)$$

where P is the consumption (W); n_s the screw rpm (min^{-1}); ρ_s the true particle density (kg/m^3); ε the packed bed voidage; d_s the diameter of the orbiting screw (m); n_a the arm rotational speed rpm (min^{-1}); l the immersion length of the screw (m).

15-12.4.2 Mix Time

$$t = \frac{k_2}{n_s}\left(\frac{1}{d_s}\right)^{1.93} \tag{15-30}$$

k_1 and k_2 are constants that must be determined experimentally on the pilot scale.

15-12.5 Scaling of Silo Blenders

The mixing action of silo blenders is due to velocity gradients generated during discharge and recirculation of material. The mixing behavior of blenders of two different sizes will be the same if they are geometrically similar (H/D ratio) and have similar velocity gradients. It has been shown in practice that homogeneity tests conducted on a 3 m^3 (volume) blender can be scaled to a 500 m^3 blender (Wilms, 1988). It should be noted that the blender internals do not always scale down with respect to blender diameter. The arching dimension (mechanical and cohesive) of a material dictates the dimensions of the internals for reliable flow.

Very few experimental data on silo blenders are available in the published literature. All manufacturers offer test facilities for pilot scale testing and use proprietary data for scale-up.

15-12.6 Specifying a Mixer

When specifying equipment for mixing of particulate solids, the following issues need to be considered:

Performance

- Metric for mixedness or expression for mixing index
- Scale of scrutiny
- Statistical limits for mixture concentration variations and acceptable frequency for concentration being outside the limits

Mechanical Issues

- *Wear:* effect of material abrasiveness on mixer internals
- *Attrition:* breakage of material during mixing process
- *Material of construction:* mixer body, seals, shafts, and gaskets
- *Headroom availability and cost:* accountability of mixer design for the cost of the structure required to house a mixer

- *Drives:* necessity for practical size of the drive and motor
- *Contamination:* material contamination due to bearings or internals
- *Cross-contamination:* need to clean the mixer between batches or product grades (especially critical for pharmaceutical processes)
- *Dust explosion:* potential for dust explosion and need to purge with inert gas
- *Hazardous materials:* need for dust or gastight containment
- *Design of internals and clearances:* depends on the potential of the particles to smear and degrade

Configuration Issues

- *Feed system:* accurate dosing system required for batch mixers, good feeder systems for continuous mixers
- *Discharge configuration:* importance of designing the system downstream of the mixer such that segregation is minimized (chutes, silos, and pneumatic transfer systems can de-mix the mixture and result in poor system performance)

After an initial selection of a suitable mixer has been made (see Figure 15-33), it is highly recommended that pilot scale testing be conducted to ensure acceptable performance upon installation.

15-12.7 Testing a Mixer

Many mixers can be operated in either batch or continuous mode. It is advised that the selection of batch versus continuous operation be made before any tests are conducted. The mixing tests should be conducted in appropriate mode. For instance, performance of a paddle mixer in continuous mode of operation cannot be reliably estimated from batch experiments.

In either case, we need to address the following questions regarding sampling and analysis:

1. Sample size
2. Location of sampling
3. Method of sampling
4. Number of samples

15-12.7.1 Sample Size. An ideal sample size is equal to the scale of scrutiny or the scale at which the product specification has been developed. If the scale of scrutiny is much smaller than the minimum amount of sample that can reliably be obtained from a sampler, suitable sample reduction techniques must be utilized. The spinning riffler has been found to be the most reliable method of reducing the sample size (Allen, 1981). The sample size must be greater than the minimum amount required for the analytical technique.

15-12.7.2 *Location of Sampling*. In practice, the most meaningful and reliable method of sampling is to take a full-stream sample for a short duration of time at the outlet of the mixer. Not only does it conform to the golden rules of sampling, this sample also accounts for any de-mixing that might occur during the discharge process.

For studying the mixing patterns within a batch mixer, the mixer must be stopped at various mixing times and sampled at various locations. These locations are usually chosen by dividing the space into equal regions. The start–stop transients during such experiments can affect the results; therefore, extended mixing without stopping must be performed for confirmation.

In the case of continuous mixers, samples must be acquired at the mixer outlet at regular frequency, starting at three times the residence time of the mixer. Usually, it is sufficient to sample up to 10 times the residence time. However, the data should also be checked for long-time scale patterns, in which case the total test time must be extended. Sampling within the mixer can be done by stopping the mixer and using sampling probes. Such data are useful in the determination of mixing length and identification of stagnant regions.

15-12.7.3 *Method of Sampling*. Sampling thieves or probes are commonly used for taking samples from stationary mixtures. It has been shown that many samplers introduce a bias by disturbing the very mixture they are supposed to sample (Figures 15-4, 15-5, and 15-6). Special designs mitigate the sampler bias. Using a scoop sample from the top surface of the mixture is the most nonrepresentative sample that one can obtain. Bias can be checked by comparing the mean composition of the samples with the composition of the entire mixture using Student's *t* test.

For continuous mixers, full-stream samplers at the mixer outlet are recommended. In-line or in situ probes to measure the physical/chemical property of interest, if available, can give useful insight into the mixing process. Before sampling, make sure that the system has reached a steady state and that at least three residence times have passed.

15-12.7.4 *Number of Samples*. Determination of the number of samples depends largely on the confidence level in the estimate of population standard deviation from sample standard deviation. The precision of the estimate of standard deviation increases with the number of samples; however, so does the analytical cost. The analytical cost is generally a small price to pay to avoid selection of an improper mixer. It is a common practice to take 15 to 30 samples during testing. As a rule of thumb, the total amount of sample removed from a batch mixer should not exceed 5% of the charge. This will prevent sample bias. Repeat tests must be conducted if the total number of samples required for analysis exceeds 5% of the charge.

15-12.8 Testing a Batch Mixer

For reliable scale-up and extrapolation of test data, it important to maintain geometric, kinematic, and dynamic similarity between the test mixer and the full scale mixer. A batch mixer should have access for sampling within the mixer at various mixing times. Batch mixers require reliable dosing measurement, which can be accomplished by weighing the components individually.

A typical batch mixer test would include the following steps:

1. Load the components in predetermined sequence. The sequence of addition of components can affect the rate at which homogeneity is achieved.
2. Mix for a known period.
3. Stop the mixer and take 10 to 15 samples from various locations within the mixer. Refer to the sampling guidelines discussed earlier.
4. Start the mixer again and repeat steps 2 and 3 at least three more times. The subsequent mixing times can be chosen in a geometric progression (e.g., 2, 4, 8, and 16 min).

15-12.9 Testing a Continuous Mixer

A continuous mixer cannot be tested reliably without a reliable feed system for the components. The feed system should be capable of turndown ratios similar to those of the full scale system. The objective of the test is threefold:

1. To determine the quality of mixture at the discharge.
2. To assess the ability of the continuous mixer to dampen feeder-related fluctuations or variance in composition.
3. To determine the residence time distribution of the components in the mixer.

The mixer operating conditions must be selected carefully based on kinematic and dynamic similarity conditions. The following experimental considerations must be heeded:

- Allow the system to reach a steady state.
- Follow the golden rules of sampling.
- Do not start sampling until three residence times have elapsed.
- Sample until about 10 residence times have elapsed or longer if necessary.
- Study start–stop transients and transients associated with feed system instabilities.

15-12.10 Process Safety in Solids Mixing, Handling, and Processing

The dust arising from the mixing or processing of combustible solid materials can form explosive mixtures with air. In the design of plants to handle such materials, consideration must be given to the potential for dust explosions and actions to prevent or minimize dust explosions. A dust explosion hazard exists when a combustible dust with particle size distribution less than 420 μm is dispersed in air or other oxidant. In general, most organic solids and metals can form explosive dust clouds. For dust to explode, certain conditions have to exist:

- Particles of dust must be of suitable size.
- Concentration of dust in the air must fall within explosive limits.
- There must be a source of ignition energy.

15-12.10.1 Important Parameters. In any unit operation involving drying, mixing, milling, conveying, storage, and so on, the following major properties of the product will probably need to be known for safe operation.

- Melt/decomposition temperature
- Potential evolution of toxic or flammable gases upon heating/combustion
- Potential for spontaneous heating
- Minimum ignition energy (MIE)
- Limiting oxidant concentration (LOC)
- Resistivity (ability to generate and accumulate static electric charge)
- Explosiveness (rate of pressure rise in closed test apparatus)
- Smoldering characteristics

15-12.10.2 General Design Safety. In addition to taking specific explosion prevention and protection steps, the conditions that can result in a secondary explosion and the frequency of ignition must be minimized. The following are important:

- Good housekeeping
- Control of mechanical sparks and friction
- Control of cutting, welding, and other open ignition sources
- Electrical bonding and grounding
- Electrical classification
- Insulating hot surfaces

15-12.10.3 Housekeeping. Good housekeeping is required for dust explosion safety in rooms or areas where dust may escape and accumulate. This dust cloud provides fuel for the secondary explosion within the room or building. Reducing significant additional dust accumulation is therefore a major factor in reducing the hazard in areas where a dust hazard can exist. A dust layer 0.8 mm thick and covering the floor of a building is sufficient to produce a uniform dust cloud of optimum concentration 3 m high throughout the building. Therefore, good housekeeping is critical for safety.

15-12.10.4 Control of Ignition Sources

Control of Mechanical Sparks and Friction. Foreign materials (such as tramp metal) that are capable of igniting combustible material being processed should be removed from the process stream by magnetic separators, pneumatic separators, or grates or other separation devices.

Control of Cutting and Welding and Open-Flame Ignition Sources. Although cutting and welding are well recognized as an ignition sources for dust explosions, these operations continue to be ignition sources

Control of Static Electricity. *Bonding* is the process of electrically connecting adjacent conductive components so that they are at the same electrical potential and no sparks can occur between them. When one or more of these components is additionally connected to an electrical ground, all of the components are then grounded, and sparks cannot occur either between them or to other systems which are also grounded. Bonding and grounding of electrically conductive components of conveying, mixing, feeding, blending, and storage systems should be provided to dissipate electrostatic buildup below hazardous accumulations. The type or extent of bonding/grounding needed is a function of the ignition sensitivity and conductivity of the material being processed or handled.

15-12.10.5 Electrical Classification. In those areas of the plant where a hazardous quantity of dust accumulates or is suspended in the air, the area should be classified and all electrical equipment and installations in those areas should comply with Article 502 or Article 503 of NFPA 70, the National Electrical Code, as applicable.

15-12.10.6 Control of Hot Surfaces. It is generally accepted that it is good engineering practice to limit the surface temperature in dust-handling or dust-processing areas to less than 80% of the minimum ignition temperature of the dust layer. Areas requiring control of hot surfaces are generally identified as those requiring Division II Electrical Classification.

15-12.10.7 Explosion Prevention and Protection. If the evaluation determines that dust explosion protection is required, dust explosions can either be prevented, or explosion venting provided, to protect against unacceptable consequences. Prevention methods are outlined in National Fire Protection Association's *Explosion Prevention Systems* (NFPA69, 2000). Explosion (deflagration) venting is outlined in *Guide for Venting of Deflagrations* (NFPA654, 2000). An outline of the general approach to protection (Eckhoff, 1991) is shown in Figure 15-59.

The following are methods of explosion protection by preventing or containing explosions:

1. Oxidant concentration reduction
2. Combustible concentration reduction

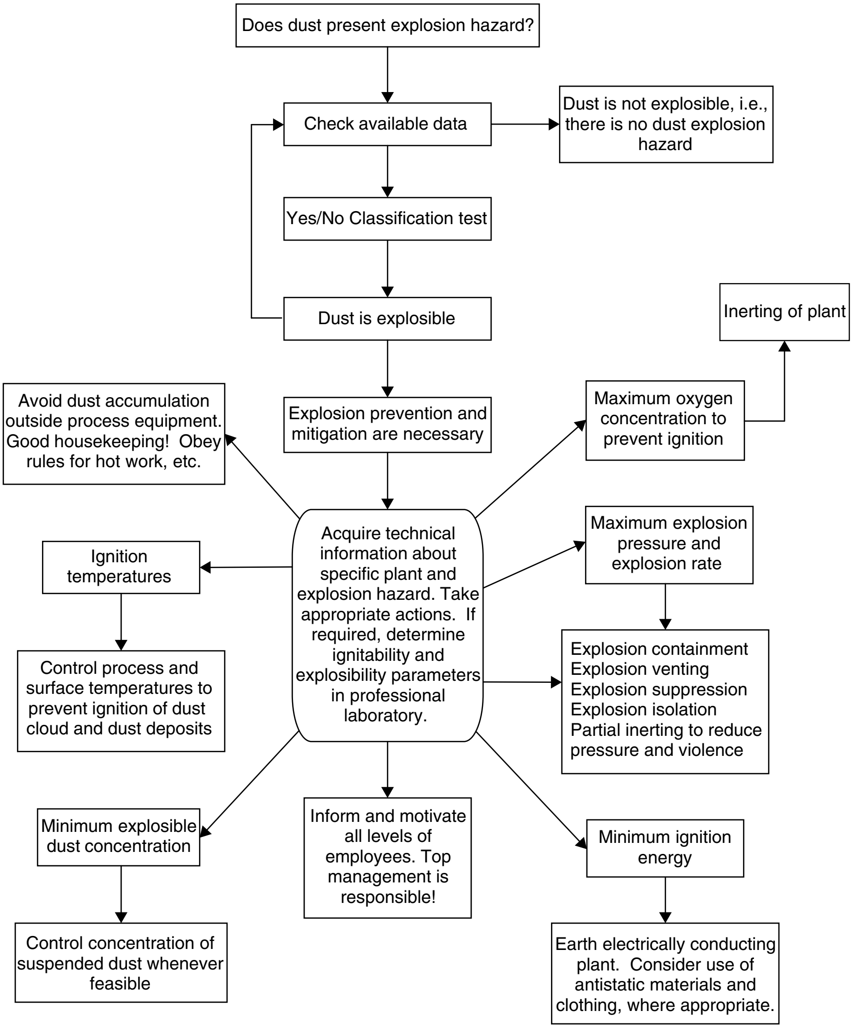


Figure 15-59 Outline of general approach to practical dust explosion protection. (From Eckhoff, 1991, modified and extended version according to Field 1982.)

3. Explosion suppression
4. Deflagration pressure containment

Methods 1 and 2 prevent combustion at a rate sufficient to result in an explosion; methods 3 and 4 are listed as protection methods based on limiting or preventing damage. Spark extinguishing is also listed as an explosion prevention method by NFPA69 (2000) but is only applicable to ducts transporting combustible dusts and must be used in conjunction with one of the other explosion prevention methods or explosion venting for protection of the complete system.

NOMENCLATURE

Part A

CoV	coefficient of variance (eq. 15-2)
I	intensity of segregation (eq. 15-1)
M	mean concentration
N_i	number of samples contained in core j
N	number of samples
RSD	relative standard deviation (eq. 15-2)
$\overline{x}$	mean composition
x_{ij}	concentration of a sample
$\overline{x_j}$	mean concentration of core *j*

Greek Symbols

σ	standard deviation
σ^2	variance of sampled data (eqs. 15-4 and 15-5)
σ_A^2	axial variance
σ_R^2	radial variance
σ_r^2	variance of randomly chosen concentration data
σ_0^2	initial variance of a fully segregated system

Part B

c_i	concentration of the ith sample
C_i	coefficient of variation of the ith component
d_s	diameter of orbiting screw (m)
D	diameter (m)
g	gravitational constant (m/s^2)
H	level of solids in hopper (m)
k_1, k_2	constants in eqs. (15-29) and (15-30)
l	immersion length of a screw in a Nauta mixer (m)
L	constant
L	mixer length (m)
m	number of samples
M	coefficient of mixing (m^2/s)
n	mixer agitator speed (rps)
n_a	screw rotation speed (min^{-1})
n_s	arm rotation speed in a Nauta mixer
N	number of particles
P	component fraction
r	mixer radius (m)
R	mixer radius (m)
s	standard deviation of sample
t	time (s)
v	peripheral velocity of the mixing element (m/s)

V	volume (m^3)
w_i	weight fraction of the i^{th} component
W	total mass (kg)
x	particle size (μm)
X	sample average
Fr	Froude number (eq. 15-20)
Ne	Newton number (eq. 15-27)
VRR	variance reduction ratio (eq. 15-14)

Greek Symbols

ε	packed bed voidage
ρ_s	true particle density (kg/m^3)
σ	standard deviation of population
ω	angular velocity (rad/s)

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CHAPTER 16

Mixing of Highly Viscous Fluids, Polymers, and Pastes

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16-1 INTRODUCTION

Viscous mixing involves the many applications in processes wherein the viscosity is sufficiently high (e.g., greater than $10\ \text{Pa}\cdot\text{s}$) that turbulent mixing is usually unobtainable, or the dissipation of the viscous energy involved would result in an unacceptably high product temperature. From simple tasks such as stirring the morning oatmeal to sophisticated industrial processes involved in manufacturing today's elastomeric and plastic compounds, recourse to laminar mixing techniques must be employed. Many industrially important products, such as pastes, putties, chewing gum, soap, grease, solid propellant, and some foods, fall into this category.

The key features that distinguish viscous mixing from nonviscous (turbulent) mixing are described in Chapters 2 and 3. In mixers handling very viscous materials, it is necessary to promote both lateral and transverse motion, with the material(s) being pulled, sheared, compressed, kneaded, and folded by the action of rotor(s) against vessel walls, saddles, or projecting stators. The condition of the feed can be an important consideration in selection of the mixer for the task. For example, a mixer for producing a uniform rubber cement solution must first cope with masticating an entire bale of rubber before starting the gradual letdown and final homogeneous operation.

16-2 VISCOUS MIXING FUNDAMENTALS

16-2.1 Challenges of High Viscosity Mixing

Mixing is an operation whereby the nonuniformity within a mixture is reduced. Mixing in very viscous systems can be a formidable task. There are no turbulent

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eddies to help distribute components. Because of the high matrix viscosity, diffusion coefficients for even very small molecules are exceedingly low. Most high viscosity fluids are also non-Newtonian; many are shear-thinning, some have a yield point. Viscous energy dissipation during mixing can cause significant temperature variation throughout a vessel, thus contributing to further viscosity nonuniformity, and possibly product degradation.

Heat transfer coefficients are also poor in very viscous systems, and the steps normally taken to improve heat transfer in cooling, such as increased agitator speed or greater temperature differences, can be counterproductive because of the heat generated from viscous energy dissipation.

The mixing task can be further complicated by the changes in state that may need to be accommodated during the process. For example, solution or homopolymerization may start out with watery thin liquids into which a very small amount of equally thin catalyst needs to be distributed uniformly, and turbines or propellers would be appropriate. However, as polymerization proceeds and viscosity starts to build, probably into the 10 to 50 $\mathrm{Pa \cdot s}$ range, these simple agitators no longer suffice, and anchor or helical ribbon mixers are more suitable. Keeping the polymerization under control means not only avoiding local hot spots but also may involve reflux cooling, so the mixing system must allow for rapid blending of the thin reflux into the viscous matrix, as well as vapor disengagement.

16-2.2 Dispersive and Distributive Mixing

Mixing can be brought about in viscous systems only by mechanical action or by the forced shear or elongational flow of the matrix. Solids with a cohesive nature (such as agglomerated particles) or other immiscible fluids (drops with interfacial tension) will require intensive mechanical stress to achieve the required dimunition. *Dispersive mixing* is defined as the breakup of agglomerates or lumps to the desired ultimate grain size of the solid particulates or the domain size (drops) of other immiscible fluids. Thus, dispersive mixing is a consequence of the history of the fluid mechanical stresses imposed on the mixture.

When mixing thermodynamically miscible fluids, or mixing hot and cold segments of the same material, mixing is determined simply by the history of deformation imparted to the fluid (the strain). *Distributive mixing* is defined as providing spatial uniformity of all the components. The interrelationship between dispersive and distributive mixing is illustrated in Figure 16-1.

In general, viscous mixing operations require some combination of dispersive and distributive actions; *intensive dispersive* mixing to break up globs or agglomerates, and *extensive distributive mixing* to apportion the broken phase throughout the co-mixture. Dispersion may be either a continuing (chronic) erosion or an abrupt (acute) rupture after surpassing a critical stress level sufficient to overcome the cohesive strength.

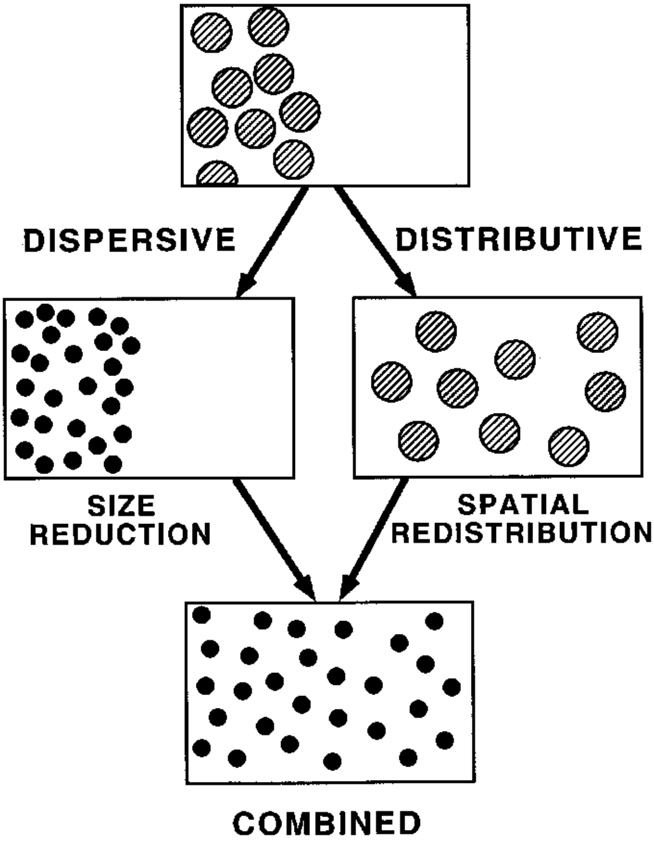


Figure 16-1 Interrelationship between dispersive and distributive mixing.

16-2.3 Elongation and Shear Flows

Convective mixing can be achieved by imposing deformation on a system through laminar flow, which can be the consequence of shear, elongation (stretching), or squeezing (kneading). Laminar flow will orient the phases, so a critical aspect of viscous mixing is to provide frequent reorientation of dispersed elements.

For *miscible* fluids (no interfacial tension), a glob or layer of A in a B matrix can undergo stretching and folding to even thinner lamella thicknesses until diffusivity, even though very low, can achieve the desired homogenization (Figure 16-2).

With *immiscible* fluids, interfacial tension (σ) will resist the forces tending to tear the dispersed phases apart. If the rheologies and concentrations are similar, the mixture could end up co-continuous, as shown in Figure 16-3. With unequal viscosities and phase ratios, the restorative action of interfacial tension (σ) will cause the stretched-out regions to break into segments (Figure 16-4). In simple shear flow, Karam and Bellinger (1968) showed that there is a maximum viscosity

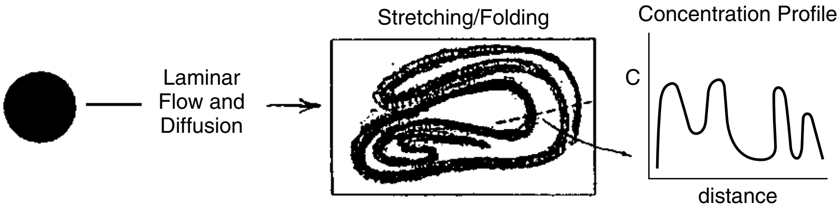


Figure 16-2 Distributive laminar mixing and diffusion with miscible fluids (no interfacial tension and very low diffusivities).

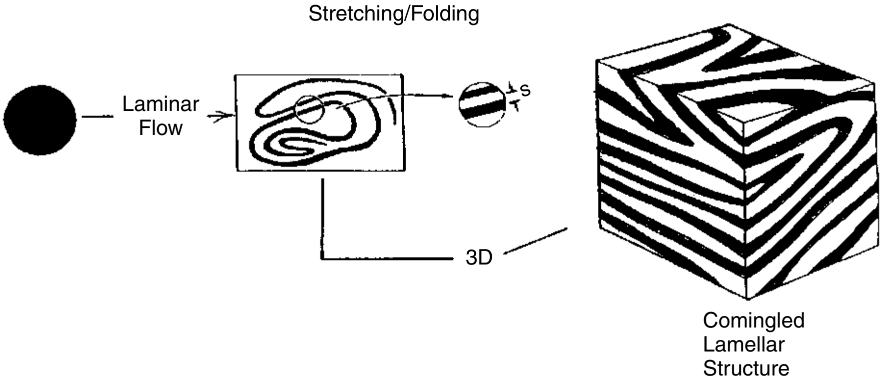


Figure 16-3 Distributive laminar mixing of immiscible fluids with similar rheologies (s is the striation thickness).

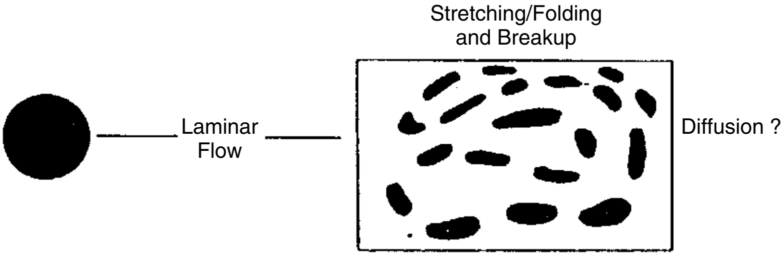


Figure 16-4 Laminar flow dispersive mixing while undergoing high stresses.

ratio, p (p equals the ratio of the dispersed phase viscosity, μ_d, to continuous phase viscosity, μ_c), beyond which a liquid droplet cannot be broken up by shear alone. Grace (1982) indicated, however, that elongational flow did not have this limitation. Figure 16-5 represents the Weber or capillary number, or the ratio of viscous forces to restorative forces, $\gamma\mu_c r/\sigma$, plotted against the viscosity ratio, p (γ is the shear rate, r is the drop radius). Based on Figure 16-5, the minimum

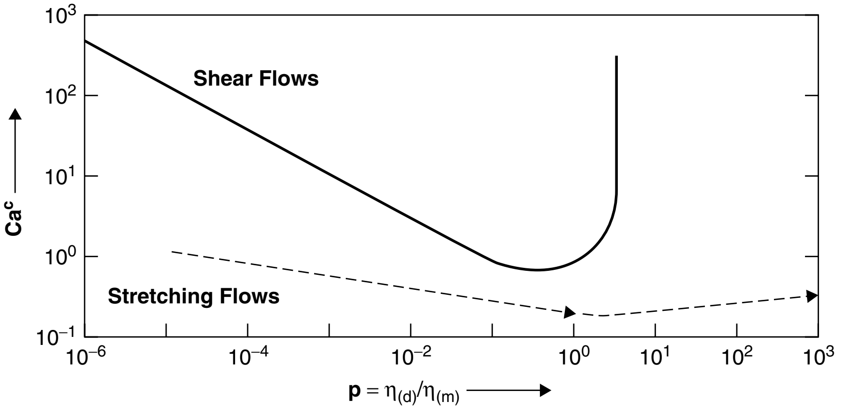


Figure 16-5 Critical Weber (or capillary) number versus viscosity ratio.

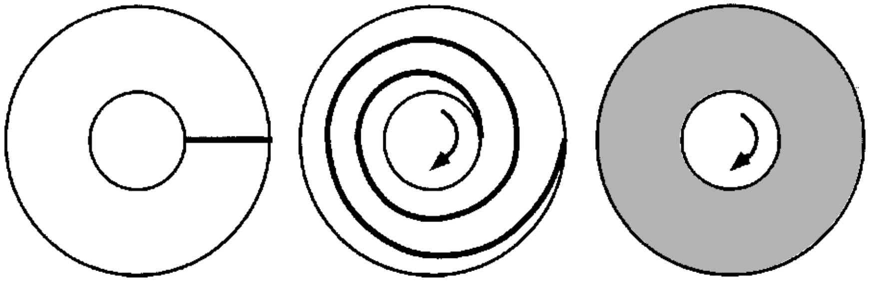
dispersed phase drop radius can be achieved where the viscosity ratio p is close to unity, but dispersion by shear flow is not possible if p exceeds 4. This limit may be different for viscoelastic fluids.

When the viscosity of a mixture exceeds 10 Pa · s, simple mixing with a conventional impeller such as a turbine or propeller stirrer will not suffice. The high viscosity may arise from a high concentration of solids in a slurry, the high viscosity of the matrix fluid itself, or by interactions between ingredients. When the viscosity is high, the mixing Reynolds number ($Re = \rho D^2 N/\mu$) is probably less than 100. As such, mixing can occur only by viscous forces, and turbulence will play no part.

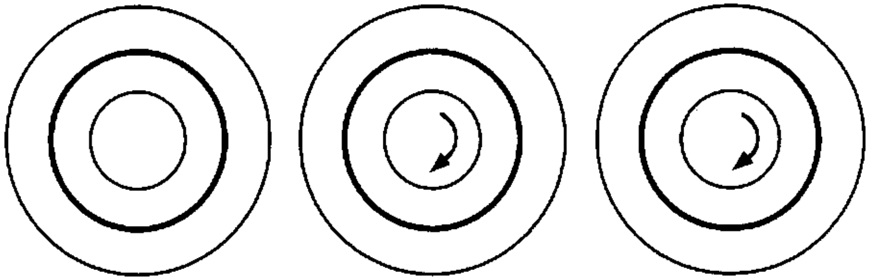
Most high viscosity mixers have a limited high-shear zone to minimize total power and heat buildup. The impellers are preferably designed to circulate all of the mixer contents past the localized high-shear zones. Particular attention must be paid to avoiding stagnant zones in batch equipment and to ensure that unmixed components are not carried through continuous mixers. Many high viscosity mixtures are shear thinning, so there may be a rapid fall-off of shear forces away from the shear-creating device. High velocity impellers may be completely ineffective since they may create an isolated cavity in the vessel without producing the required circulation (see Figure 18-13 and Chapter 9).

The basic requirement for accomplishing mixing in viscous systems is deformation of the mixture. Simple shear is inefficient and insufficient. Additional complexity must be incorporated into the system. The inefficient orientation of simple shear must be disrupted. When dispersion is also required, region(s) of intense deformation must be created by having flow forced through narrow passageways either through passive orifices (consuming pressure drop) or between walls which move with respect to each other, such as provided by a closely fitting impeller (consuming power for rotation).

The critical effect of orientation is shown in Figure 16-6, wherein the inner cylinder rotates with respect to the outer cylinder. If placed initially



INITIAL ORIENTATION (RADIAL) (*a*) AFTER TWO TURNS (*b*) AFTER MANY TURNS (*c*)



INITIAL ORIENTATION (CONCENTRIC) (*d*) AFTER TWO TURNS (*e*) AFTER MANY TURNS (*f*)

Figure 16-6 Effect of orientation on mixing in a concentric cylinder mixer.

radially between the two cylinders, the interface, increases linearly with time (Figure 16-6*a–c*), but with an ever-decreasing striation thickness. If the interface is originally also concentric, as in Figure 16-6*d*, the striation thickness remains constant regardless of the amount of rotation. A video clip of this process is provided on the Visual Mixing CD affixed to the back cover of the book.

16-2.4 Power and Heat Transfer Aspects

16-2.4.1 Power. For equipment wherein the agitator sweeps near the vessel wall, the power drawn is due primarily to viscous drag rather than from the pumping required for circulation. The power for a Newtonian viscous mixer evolves thus:

shear rate = $\alpha DN/t$
shear stress = (shear rate) (viscosity) = $\alpha DN\mu/t$
shear area = α DL = α DL
force = (shear stress) (area) = $\alpha D^2LN\ \mu/t$
torque = (force) (radius) = $\alpha D^3LN\ \mu/t$
power = (torque) (speed) = $\alpha D^3LN^2\mu/t$

On scale-up, with dimensional similarity of both blade length L and clearance t proportional to diameter,

$$\text{power} = \alpha\mu D^3N^2 \tag{16-1}$$

For very viscous mixtures, the viscosity is likely non-Newtonian. For shear-thinning fluids that can be represented by a power law exponent (n);

$$\text{power} = \alpha\mu D^3N^{1+n} \tag{16-2}$$

16-2.4.2 Heat Transfer. Correlations that have been developed for heat transfer in wiped vessels usually take the form of the dependence of Nusselt number (hD/k) on Reynolds number ($D^2N\rho/\mu$), and Prandtl number ($C_p\mu/k$), and include a slight dependence on viscosity ratio (μ/μ_w), where μ_w is the viscosity at the wall:

$$\text{Nu} \propto \text{Re}^a\text{Pr}^b\left(\frac{\mu}{\mu_w}\right)^c$$

Figure 16-7 shows correlations for the dependence of heat transfer on Reynolds number for a variety of such vessels. As the viscosity increases, dependency on Reynolds number, exponent *a*, shifts from $\frac{2}{3}$ to $\frac{1}{3}$ (Figure 16-7). A selection of correlations for vessels is given in Chapter 14. In the Reynolds number range 10^{-3} to 10, Todd (1988) correlated data for twin-screw extruders as follows:

$$\text{Nu} = 0.94\text{Re}^{0.33}\text{Pr}^{0.33}\left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{16-3}$$

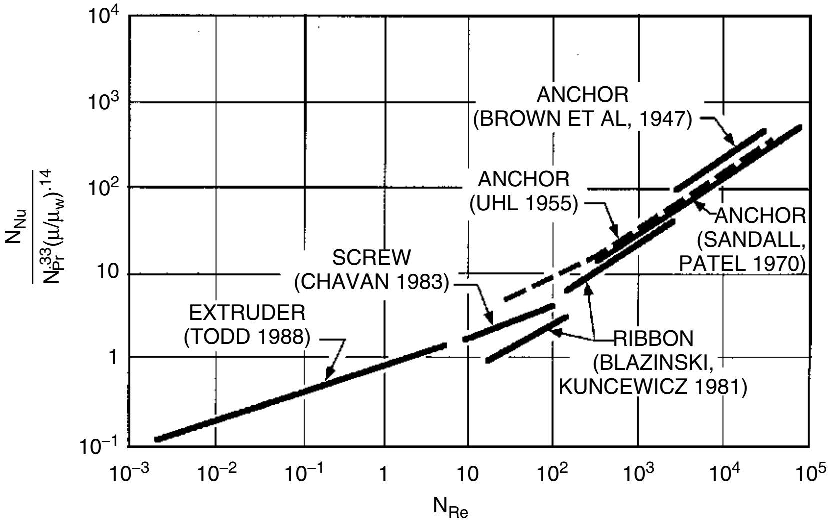


Figure 16-7 Nusselt–Prandtl–Reynolds number correlations for heat transfer.

16-3 EQUIPMENT FOR VISCOUS MIXING

Characteristics of mixers for viscous mixing may include:

- Small clearances between impeller and vessel walls
- High power per unit volume
- Relatively small volume
- Slow impeller speeds to limit heat buildup
- Smearing blade profile if dispersion is important
- Scraping profile if heat transfer is critical
- Intermeshing blades or stators to keep the material from cylindering on the rotating impeller
- Special consideration for emptying

16-3.1 Batch Mixers

16-3.1.1 Single-Stirrer Mixers. Viscous mixtures such as thick pastes or polymer solutions can be handled in a batch mixer as long as the agitator is in close proximity to the vessel wall. The two most common types are anchor blades (Figure 16-8) and helical ribbons (Figure 16-9). The latter are generally preferred because they provide for end-to-end axial turnover as well, and usually require less power than anchor mixers. Since very viscous mixtures are frequently shear thinning, the goal of achieving effective flow throughout the vessel usually precludes the use of propellers or turbine blade agitators, which may merely spin

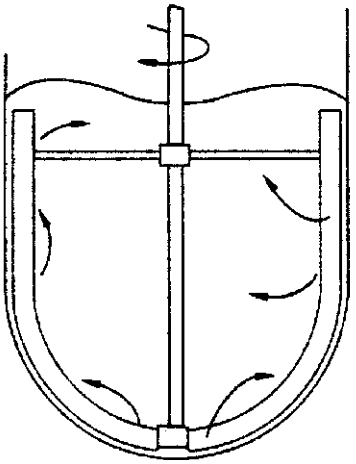


Figure 16-8 Anchor mixer. A video clip of mixing with the anchor mixer is provided on the Visual Mixing CD affixed to the back cover of the book.

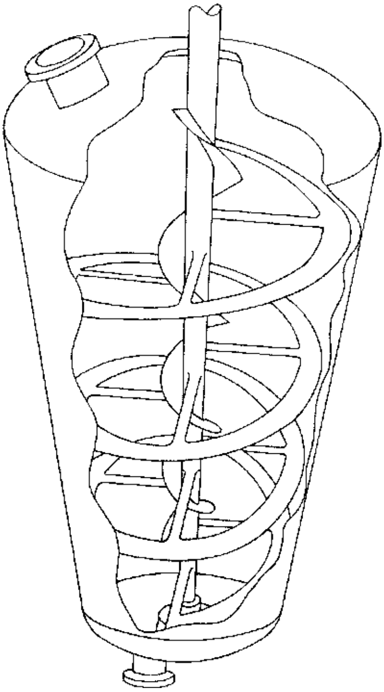


Figure 16-9 Helical blade mixer. A video clip of blending with a helical ribbon is provided on the Visual Mixing CD affixed to the back cover of the book.

in a central zone without causing any motion at the vessel walls. This is discussed at more length in Chapter 9.

Bakker and Gates (1995) provide some guidelines for power and blending time, and specifically compare a helical ribbon against a turbine impeller for an intermediate viscosity (25 Pa · s) application (Re = 90). For the same blend time (4.5 min), the turbine impeller would require over four times as much power.

Carreau et al. (1993) determined power consumption for six helical ribbon–tank combinations differing in diameter, impeller pitch, and blade width, and with various test fluids. In the laminar region ($Re < 50$) the power number (N_p) for Newtonian fluids could be correlated by

$$N_p = \frac{K_p}{Re}$$

with K_p ranging from 120 to 192 for the six combinations. Carreau et al. (1993) indicate that the effective shear rate [based on the Metzner and Otto (1957) correlations] is strongly dependent on the rheological characteristics of the fluid being mixed, but only weakly dependent on agitator geometry.

With very viscous mixers, the time to empty the vessel may also be an important consideration. Significant degradation may occur if fluid remains stuck to the tank walls and internals. Again, the helical ribbon mixer would be the preferred option for fixed installations.

16-3.1.2 Change Can Mixers. These mixers allow for separation of the mixing blade(s) from the mixing vessel, thereby providing more accurate weighing of ingredients prior to mixing, less batch-to-batch cross-contamination, easier cleaning, and less tie-up of the mixer while the tank is being emptied. Frequently, agitation is provided by planetary mixing blades (Figure 16-10). Change can mixers are available over a wide range of sizes from 1 L to 4 m^3.

16-3.1.3 Double Arm Kneading Mixers. As illustrated in Figure 16-11, these mixers have two horizontally mounted mixing blades, either tangential or slightly overlapping. The bottom of the trough consists of two half-cylinders that match the sweep of the mixing blades. Various blade shapes have evolved, as

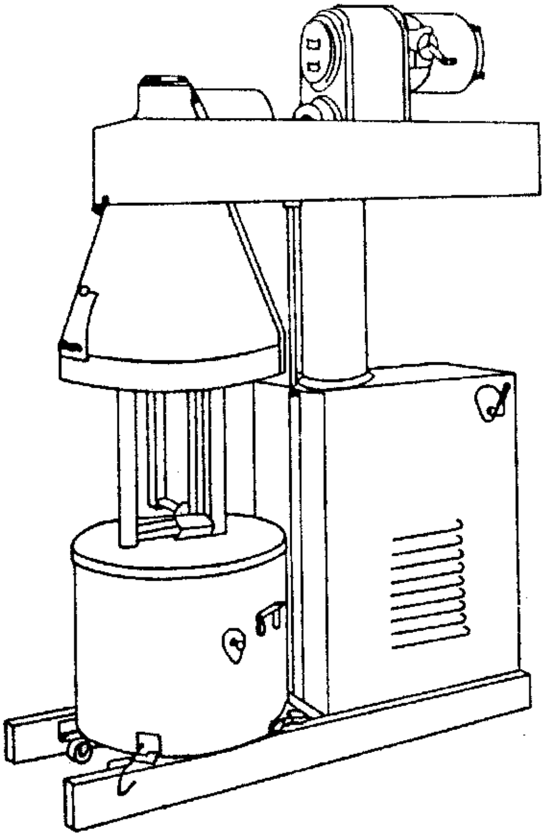


Figure 16-10 Change can mixer.

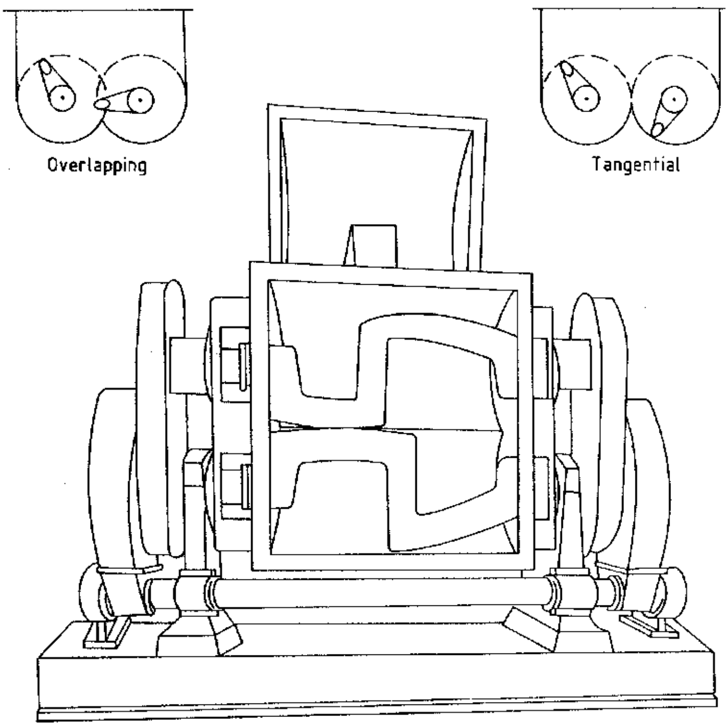


Figure 16-11 Double arm kneading mixer.

shown in Figure 16-12. The most common is the Sigma-blade (Figure 16-12*a*), with unequal wings to induce randomness.

With tangential blades, additional randomness in introduced by having the blades turning at different speeds. Using acid–base titration in a viscous non-Newtonian CMC (carboxymethyl cellulose) solution, the time for complete mixing was determined as a function of blade speed ratio, as shown in Figure 16-13, where equal rotational speeds are shown to take almost twice as long to reach molecular scale mixing as when the blades are turning at different speeds (e.g., a speed ratio > 1.2). For Newtonian and many non-Newtonian mixtures, the time to achieve molecular scale mixing is generally inversely proportional to average blade speed, as shown in Figure 16-14.

One of the problems in scale-up is that viscous energy dissipation is usually great with high viscosity fluids, yet the surface/volume ratio decreases with increasing size. At the same mixer speed, the torque and dissipation are higher in a high viscosity fluid. Scale-up at constant dissipation requires decreasing N as $N_L/N_S = (D_S/D_L)^{3/2}$, but the area per volume that is available for heat transfer decreases faster, with D_S/D_L. Consequently, most mixer manufacturers decrease blade speed and power/volume as size is increased, to prevent product degradation. This results in longer mixing times at the large scale.

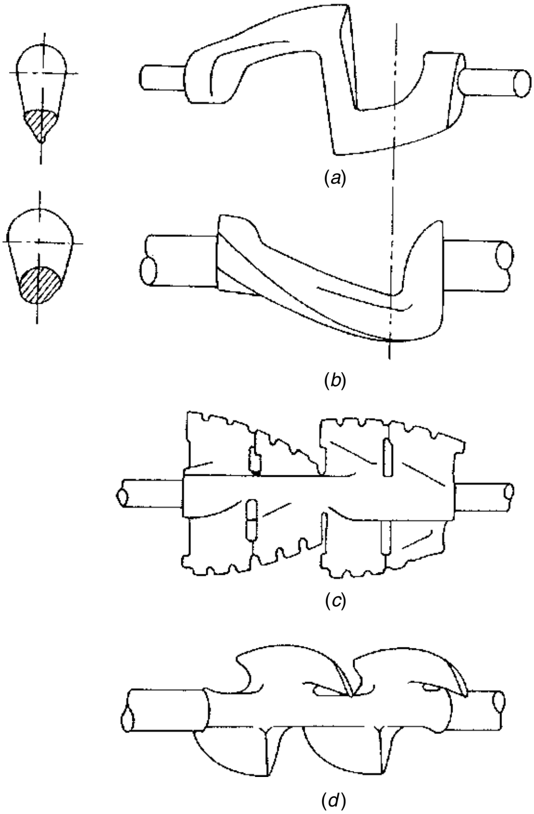


Figure 16-12 Agitator blades for double arm kneaders: (*a*) Sigma; (*b*) dispersion; (*c*) multiwing overlap; (*d*) double Naben.

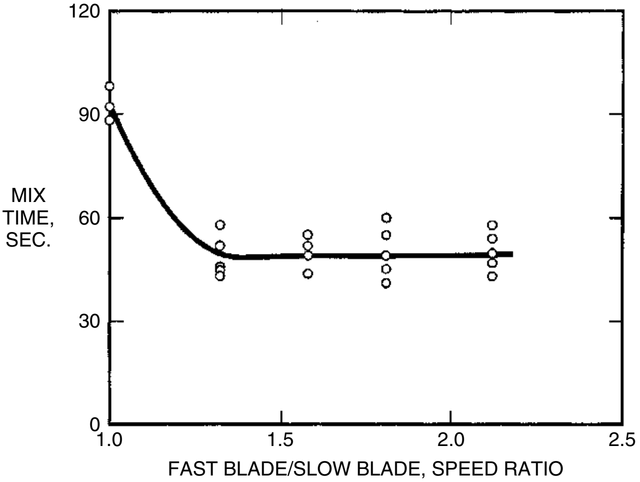


Figure 16-13 Effect of blade speed ratio on mixing time.

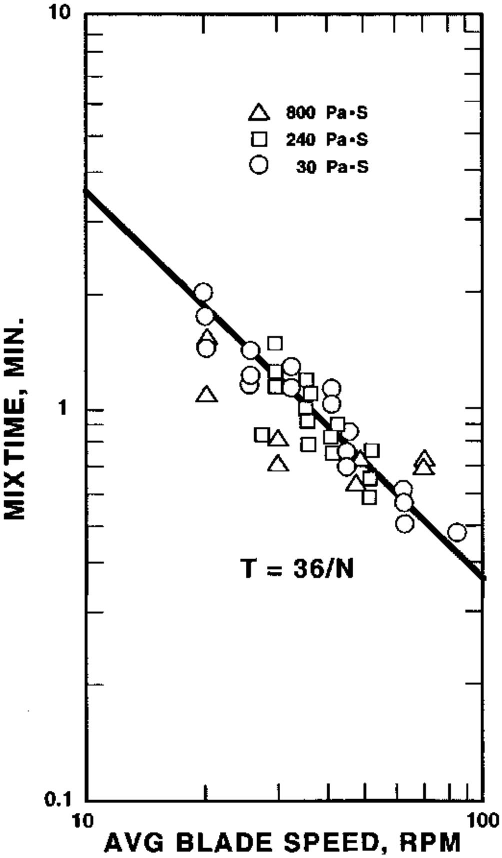


Figure 16-14 Mixing time in a 9 L Sigma-blade batch mixer.

The Sigma-blade mixer may be tilted for discharge, be equipped with a bottom discharge door, or contain an auxiliary screw discharge, as shown in Figure 16-15. In the latter case, the screw is rotated inward during the mixing cycle to provide additional mixing action, and then reversed for discharge. Sigma-blade mixers range in size from 1 L to 5 m^3. Power inputs range from 0.02 to 0.5 kW/kg, as shown in Figure 16-16 for typical applications (Parker, 1965).

The Banbury mixer (Figure 16-17) is a very high power (up to 6 kW/kg) batch mixer equipped with a top ram to force material into the mixing zone, and with bottom discharge when the batch cycle is complete. Because of the short L/D and with the shafts supported at both ends, this mixer is frequently used for very viscous materials such as rubber.

Plow mixers (Figure 16-18), ribbon blenders (Figure 16-19), cone and screw mixers (Figure 16-20), and Mullers (Figure 16-21) are used for free-flowing paste mixing applications (as well as just for solids blending purposes) where the power requirements are not too high.

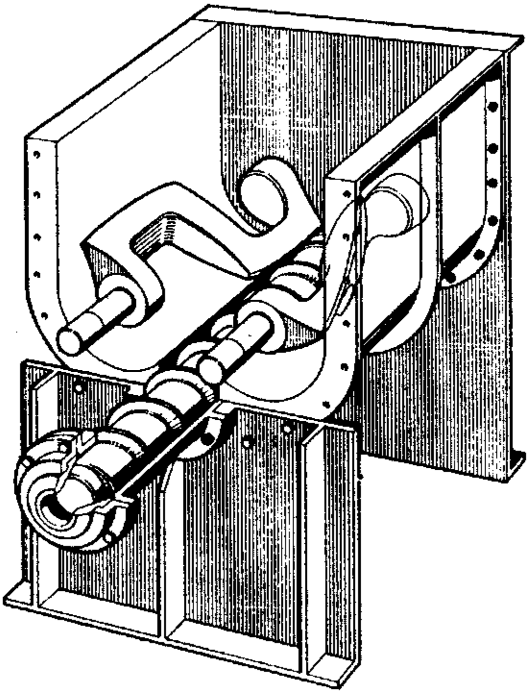


Figure 16-15 Screw discharge batch mixer.

16-3.2 Continuous Mixers

Although batch mixers could perhaps be converted to continuous operation, they rarely are because the broad residence time distribution would lead to product nonuniformity. Continuous mixers require accurate metering of ingredients. Usually, one or two rotors operate in an open or closed trough, which may be jacketed. In larger units, the rotors may be cored to provide additional heat transfer area. The rotor(s) may have interrupted flights to interact with pins or baffles projecting inward from the trough wall to improve blending by the action of flow division and recombination. The rotors usually are in close proximity to the trough or barrel wall. A restriction may be placed at the discharge end to control holdup, residence time, and mixing energy.

16-3.2.1 Single-Screw Extruders. A necessary step in most polymer processing is melting of the plastics to be able to homogenize mixtures incorporating stabilizing agents, color, fillers, and so on. Melting cannot be accomplished by direct heat transfer because of the inherently low thermal conductivities of most polymers, and too great a temperature difference driving force at the vessel wall will lead to scorching and product degradation.

Single-screw extruders (SSEs) convert mechanical energy of the drive into thermal energy, most of which is utilized in melting the feed polymer. The

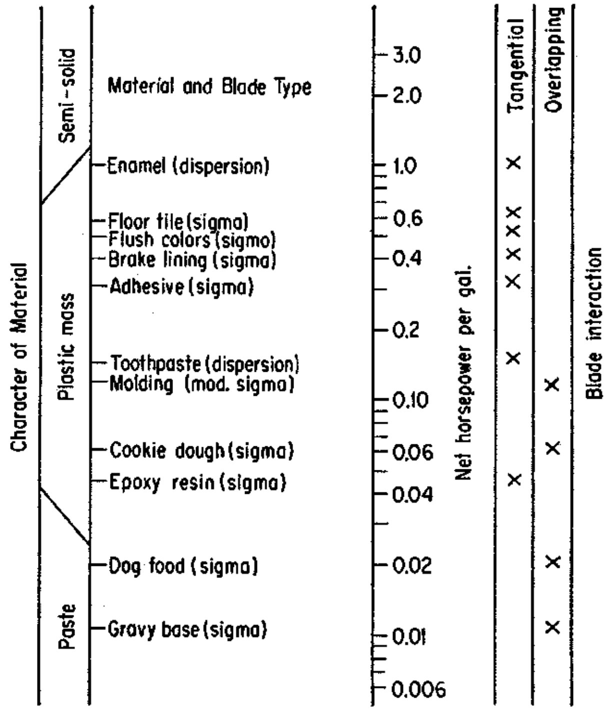


Figure 16-16 Typical applications and power for double arm kneading mixers.

enthalpy of some common polymers is shown in Figure 16-22 relative to a datum at 20°C. SSEs enable the processor to combine melting, mixing, off-gassing, and developing pressure for product shaping (profile, film, sheet, pellets).

A basic SSE is shown in Figure 16-23. Solid feed, as powder, granules, or pellets, is generally flood-fed to the extruder, wherein the feed rate is controlled by the takeaway capacity of the rotating screw. An SSE generally has a continuous helical flight, typically with a lead, Z (length for 360° turn), about equal to the diameter, D, and a channel depth, h. The root diameter must be large enough to handle the torque.

Melting occurs by friction of the solids being moved forward by the screw against the close-fitting barrel wall as the channel depth decreases through a transition zone between the feed and the metering section. As melt forms, viscous energy dissipation becomes the predominant energy transfer mechanism.

The metering section acts as a melt pump, dragging the melt forward. Since sufficient pressure will need to be generated to overcome the resistance of any filter or shaping die at the discharge end of the extruder, the molten mixture will want to flow back upstream, offsetting some of the drag flow.

The net capacity of an extruder (Q) for pumping a viscous melt is expressed as

$$Q = Q_d - Q_p \tag{16-4}$$

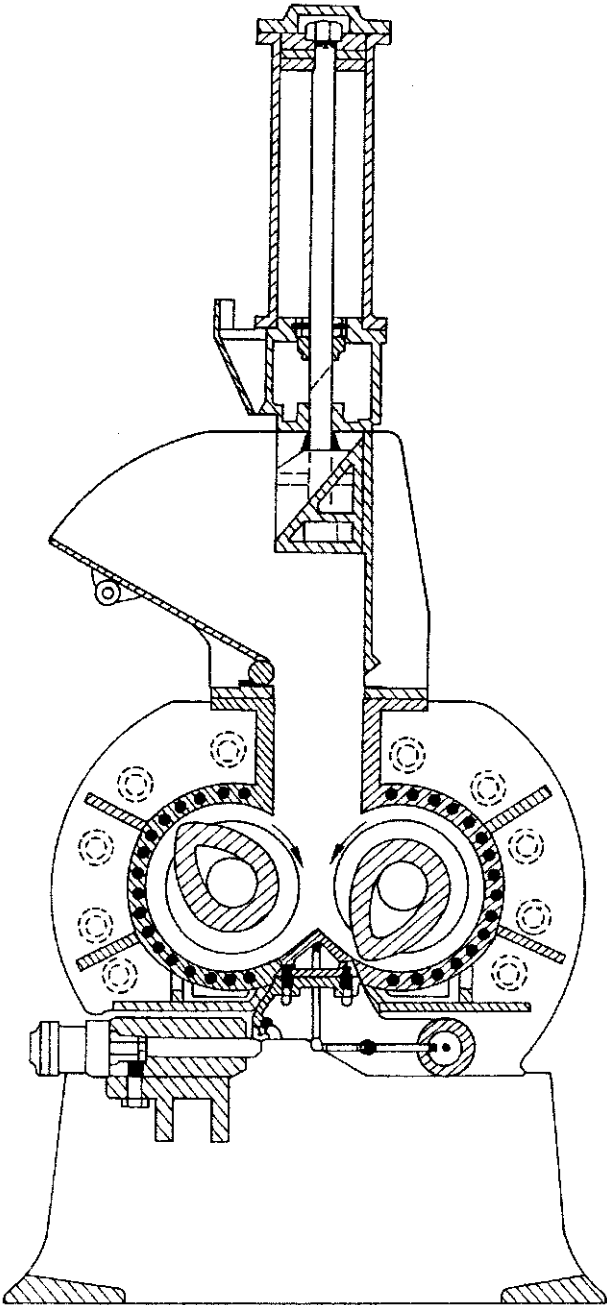


Figure 16-17 Banbury mixer.

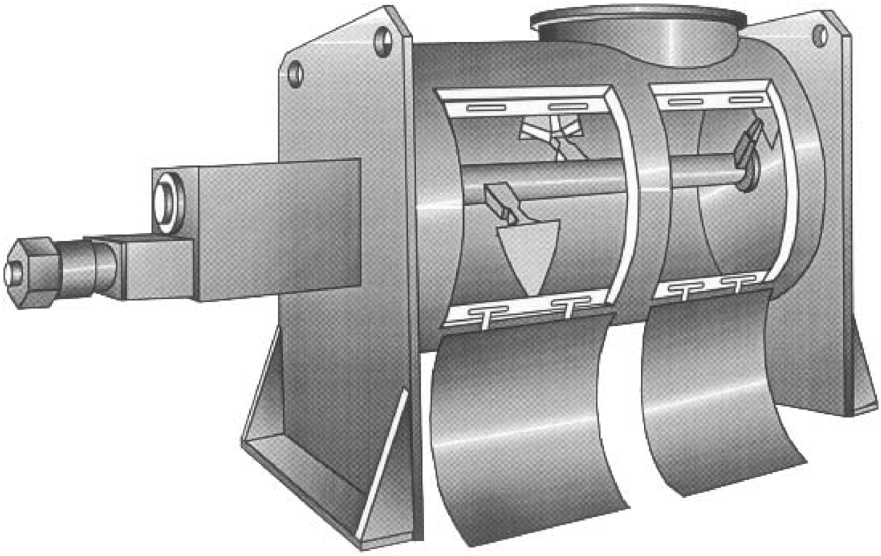


Figure 16-18 Plow mixer.

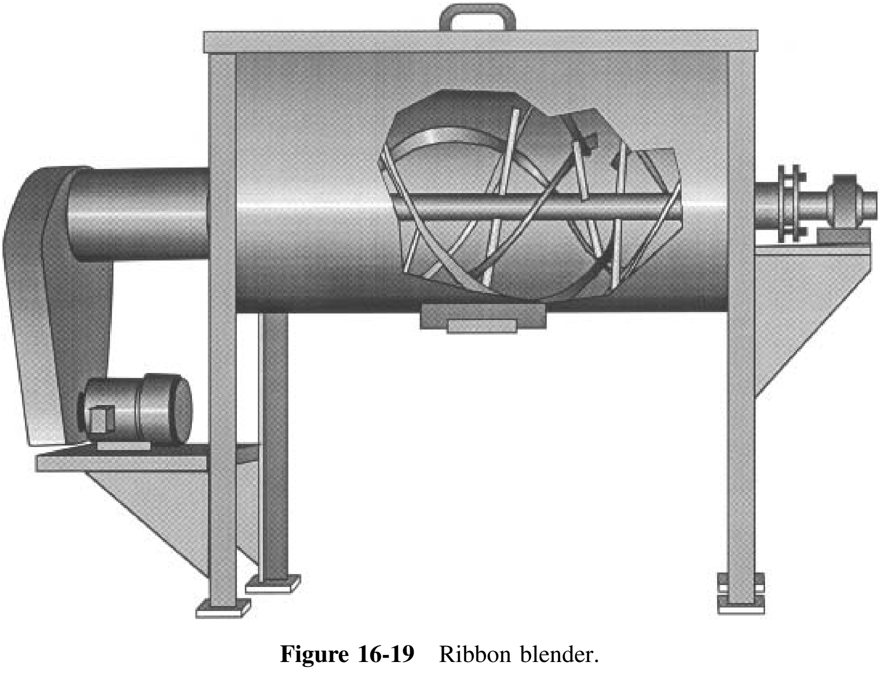


Figure 16-19 Ribbon blender.

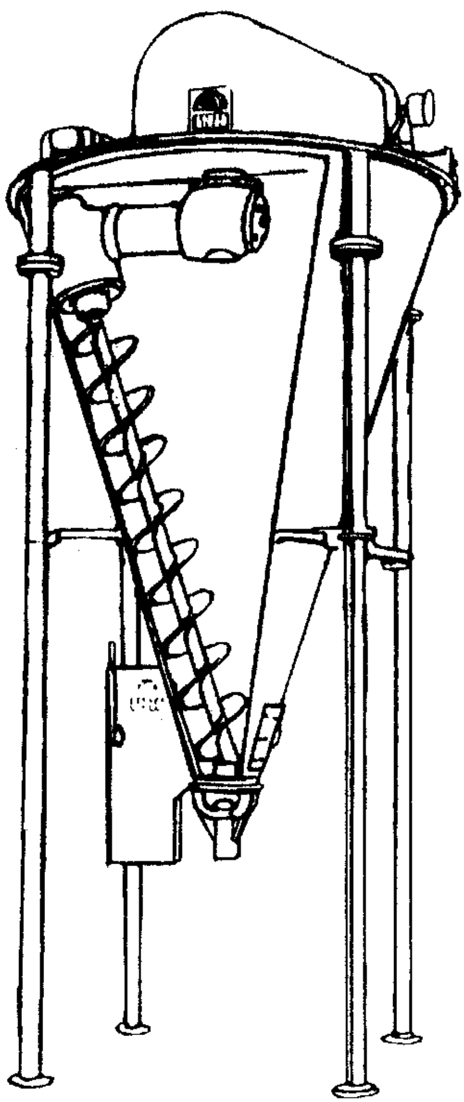
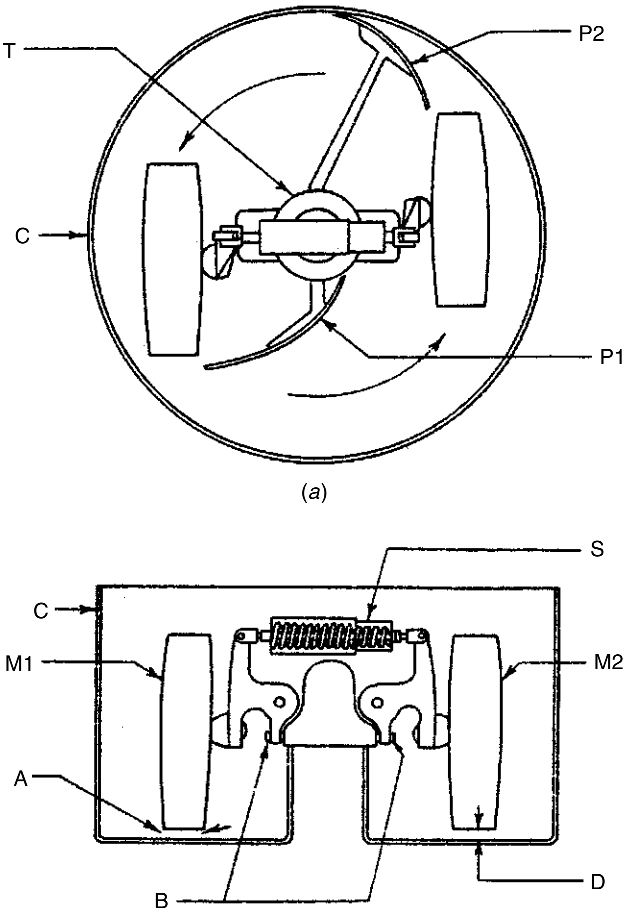


Figure 16-20 Cone and screw mixer.

where Q_d is the volumetric drag flow forward and Q_p is the volumetric pressure flow back down the channel.

$$Q_d = aN \tag{16-5}$$

$$Q_p = \frac{b\Delta P}{\mu L} \tag{16-6}$$



(*a*)

(*b*)

Figure 16-21 Muller.

$$a = \frac{F_d \pi D W h \cos\theta}{2} \tag{16-7}$$

$$b = \frac{F_p W h^3 \sin\theta}{2} - e \tag{16-8}$$

$$W = \frac{\pi D \sin\theta}{2} - e \tag{16-9}$$

where F is the shape factor dependent upon h/w, D is the screw diameter, W the channel width, h the channel depth, θ the helix angle, and e the flight width. Textbooks such as Tadmor and Gogos (1979), Rauwendaal (1990) and Chung (2000), provide many details on the workings of SSEs.

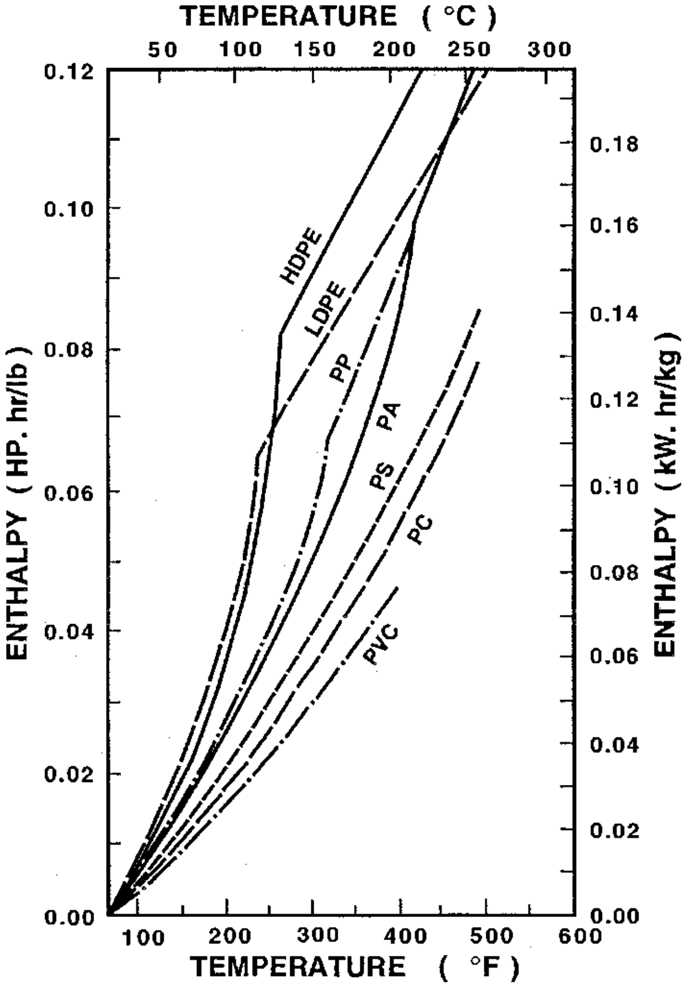


Figure 16-22 Enthalpy of common polymers (enthalpy = 0 at 20°C).

Solid additives may be dry blended in the desired ratio in a ribbon blender or intensive mixer upstream of the extruders. Alternatively, it may be important to melt the polymer fully before solid fillers are added downstream, particularly if a large volume is to be incorporated. In the act of melting pellets in the presence of powdered fillers, compressive forces may be created that can actually cause agglomeration, akin to briquetting. The agglomerates so formed may then survive into the final product. Postponing filler addition until after the base resin is fully molten will also lessen barrel and screw wear. Downstream addition of filler will require proper venting of the air that accompanies introduction of low-bulk-density powders. The merits of alternative modes of incorporating solids are described by Todd (2000).

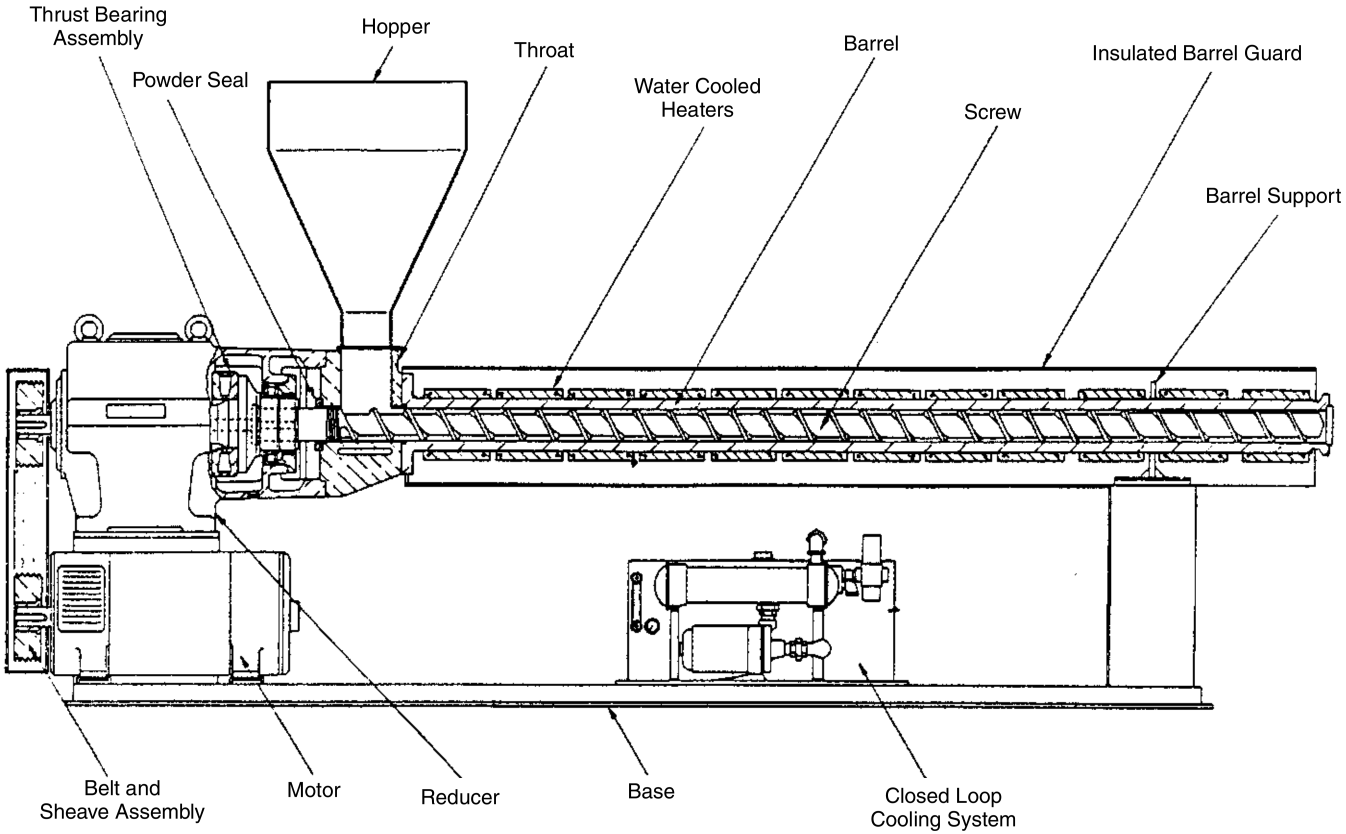
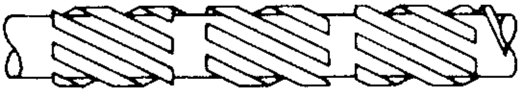


Figure 16-23 Single-screw extruder.

If venting is required, a two-stage extruder is used, with an intermediate deep channel zone following the first metering zone. A melt seal is created at the end of the metering zone to prevent sucking gases from the feed port. After transit through the vent zone, the channels are only partly filled and the volatile material is removed through the vent port in the barrel. The melt mixture passes through a second metering zone to develop the required discharge pressure.

16-3.2.2 Mixing Enhancers. Cross-channel flow occurs in the full flights of an extruder due to the combined actions of drag and pressure flows. Frequently, most of the mixing may be accomplished by the time that the last bit of polymer has been melted. Various devices have been employed to provide final homogenization before discharge, such as those illustrated in Figure 16-24.

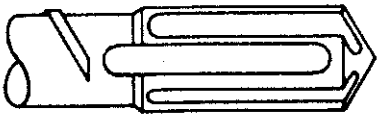
The Maddock mixing section forces flow over a narrow clearance between inlet and outlet flutes. This device not only forces all the product through a high-shear zone but can also act as a crude filter to prevent the passage of gross agglomerates downstream. Most of the other mixing promoters shown depend on generating elongational flow patterns and multiple dividing and recombining of the split flows (as well as localized high-shear zones). As such, they also require an additional pressure driving force, but the latter involves less energy than the original melting.



Parallel interrupted mixing flights

Ring barrier

Mixing pins



Maddock mixing section

Figure 16-24 Mixing enhancers for single-screw extruders.

Generating pressure in an extruder is not thermodynamically efficient, and extruders may be only about 10% efficient (E) as a pump. The adiabatic temperature rise (ΔT) accompanying pressure rise (ΔP) for an extruder is:

$$\Delta T = \frac{\Delta P}{E\rho C_p} \tag{16-10}$$

With a typical specific gravity (ρ) equal to unity, a melt heat capacity C_p of $0.5\ \text{cal/g}\cdot{}^\circ\text{C}$, and 10% efficiency (E):

$$\Delta T(^\circ C) \approx \frac{\Delta P(\text{bar})}{2} \tag{16-11}$$

For example, following venting in a two-stage extruder, the adiabatic temperature rise accompanying generation of a 1450 psi (100 bar) discharge pressure would be about an additional 50°C.

The Kokneader (Figure 16-25) is a special single-screw extruder with interrupted flights on the screw and mixing teeth projecting inward from the barrel wall. The screw reciprocates as well as rotates. Passage of the teeth through the channels creates multiple mixing actions, as described by Case (1998), as well as preventing adherence of the mixture within the screw channel.

16-3.2.3 Twin-Screw Extruders. Single-screw extruders take advantage of the interaction between screw and barrel. Twin-screw extruders (TSEs) capitalize on the interaction between the two screws. TSEs can be classified as being counterrotating or co-rotating, and tangential or intermeshing (accepting also varying degrees of intermesh). The three types of commercially available TSEs are shown in Figures 16-26 and 16-27 (corotating tangential designs are not offered). In polymer processing, TSEs perform the full gamut of solids feeding, melting, mixing, reacting, venting, and pressure development (viscous pumping). How the various functions are performed in representative types of commercially available equipment is fully described in a book edited by Todd (1998). We concern ourselves here primarily with the mixing function.

In all extruders being fed solids, some mixing may occur simultaneously with melting. However, more reliance is placed on creating flow fields within the screw channels to enhance both strain and elongational stresses conducive to dispersive and distributive mixing. For *nonintermeshing counterrotating TSEs* (Figure 16-26), the flow within each screw's channel is similar to that in SSEs, with additional reorienting as flow oscillates between the screws when in a staggered array. As with SSEs, the screw channels effectively achieve mixing only when full, so blister rings or reverse helix screws can be artfully placed to dictate the degree of fill. Nonintermeshing counterrotating screws can provide good distributional mixing, but are not particularly effective in dispersive mixing. An excellent summary of this type of TSE is given by Bash (1998).

Another type of nonintermeshing rotor mixer is the continuous mixer, such as that shown in Figure 16-28, which is in essence a continuous version of the

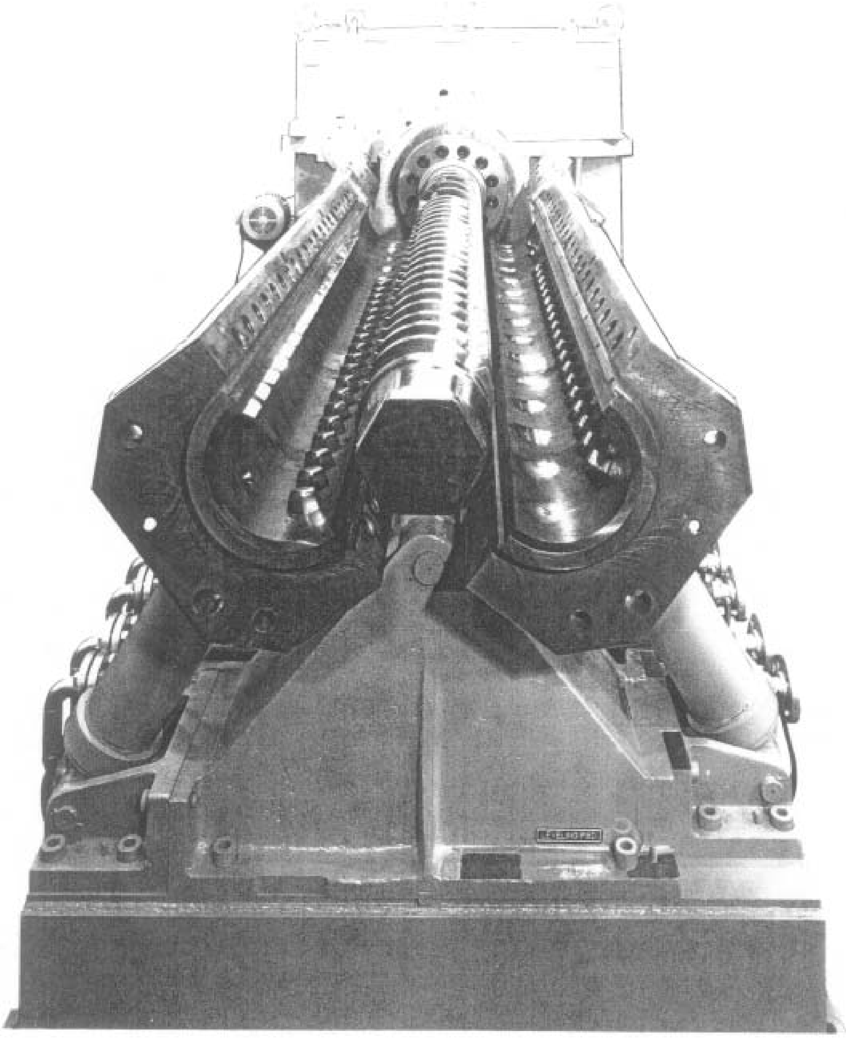


Figure 16-25 Reciprocating single-screw extruder (Kokneader).

Banbury mixer (Figure 16-17). Again, the short L/D shafts are supported at each end, and very high intensity mixing is possible (Canedo and Valsamis, 1998). Discharge is radial, with a variable restriction to control the holdup. A separate melt extruder is used to develop pressure for pelletizing.

Traditional intermeshing counterrotating TSEs, shown in Figure 16-29, consist of a series of essentially closed off C-shaped chambers (Figure 16-30) that march down the barrel with little interchange between chambers except that which occurs by leakage, as shown by Janssen (1978). There is a milling effect, not unlike that of a two-roll mill, with good elongational flow and good dispersion

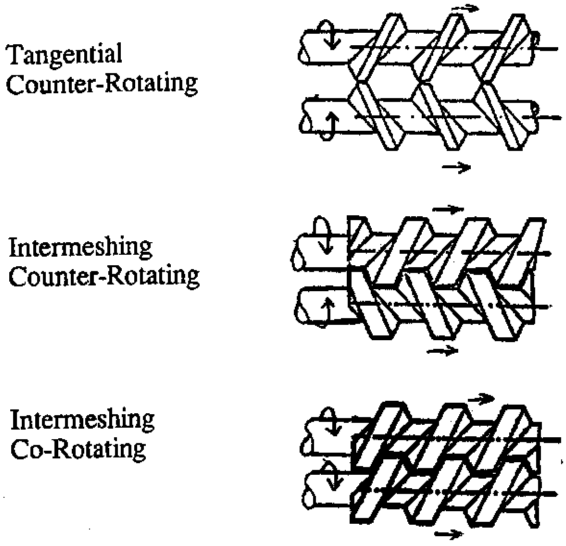
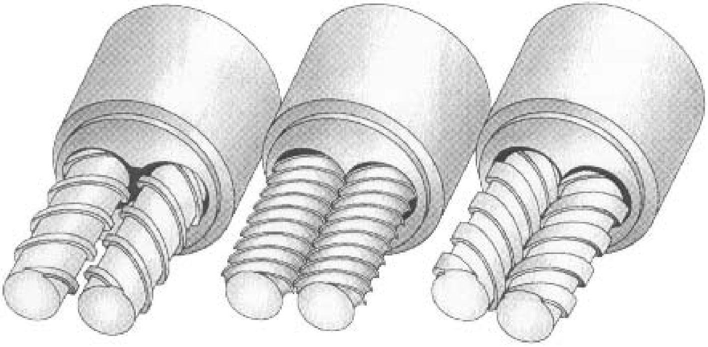


Figure 16-26 Classification of twin-screw extruders.



Tangential counterrotating nonintermeshing

Fully wiping co-rotating intermeshing

Fully calendering counterrotating intermeshing

Figure 16-27 Classical formats of twin-screw compounding extruders.

between the flights of one screw in cooperation with the channels of the other. With almost half of each screw in close contact with the barrel, the conventional intermeshing counterrotating TSE has regions of low volume undergoing high shear and potential overheating, thereby limiting the screw speed and potential capacity of this type of TSE. Thiele (1998) has described the mixing action that can be achieved with lobe-type mixing elements, as depicted in Figure 16-31. These elements can be multilobed to produce a great variety of flow patterns conducive to enhanced mixing, both dispersive and distributive.

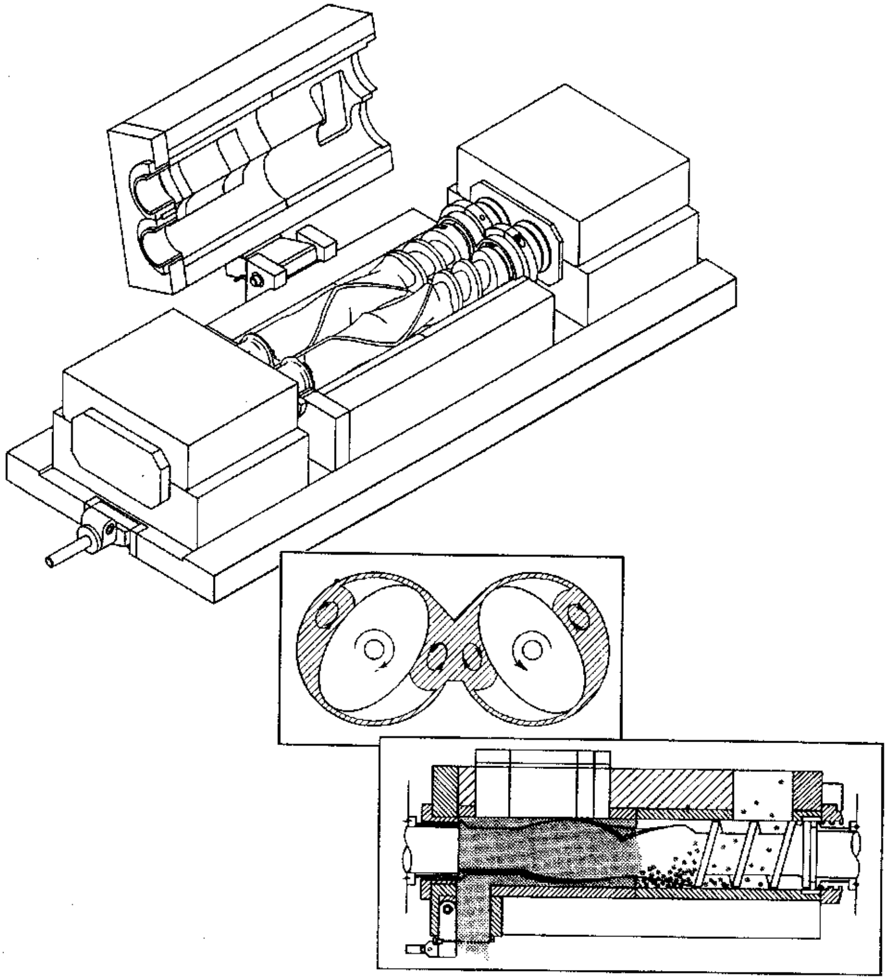


Figure 16-28 Farrel continuous mixer.

The most common variety of TSE is the intermeshing corotating type, shown in Figure 16-32. Flight tips on one rotor intermesh and wipe the channel of the other. The main mixing action, however, arises from the use of kneading paddles, which can generate a mixing action not available in the other types of TSEs. The kneading paddle has the same cross-section as the screw, with the same self-wiping feature. The most common configuration for mixing is a bilobe design (Figure 16-33), but single- and trilobe designs also exist (Figure 16-33). The kneading paddles are frequently grouped in units of three to five for mechanical strength, to form kneading blocks of fixed angular offset and with a variety of axial lengths. The kneading blocks may have some of the conveying characteristics of screws, depending on paddle width and offset, as shown in Figure 16-34.

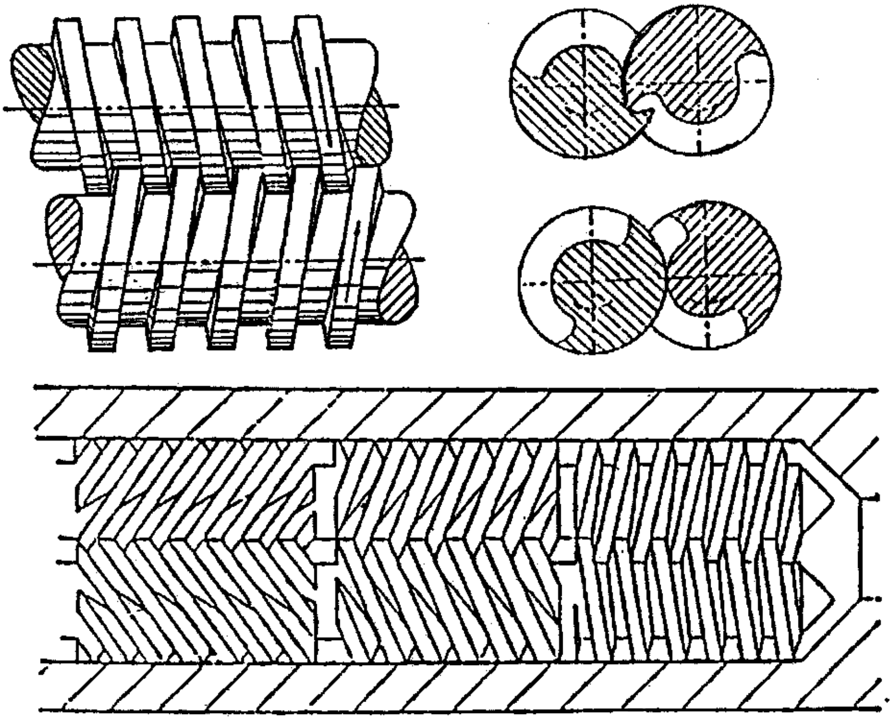


Figure 16-29 Counterrotating intermeshing screws.

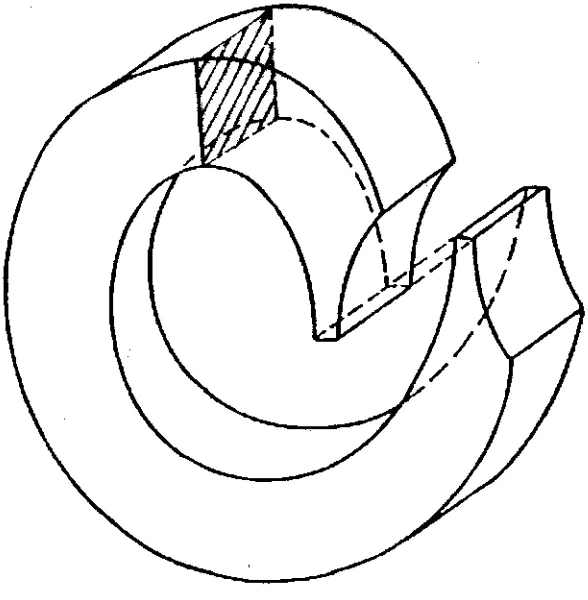


Figure 16-30 C-shaped channel.

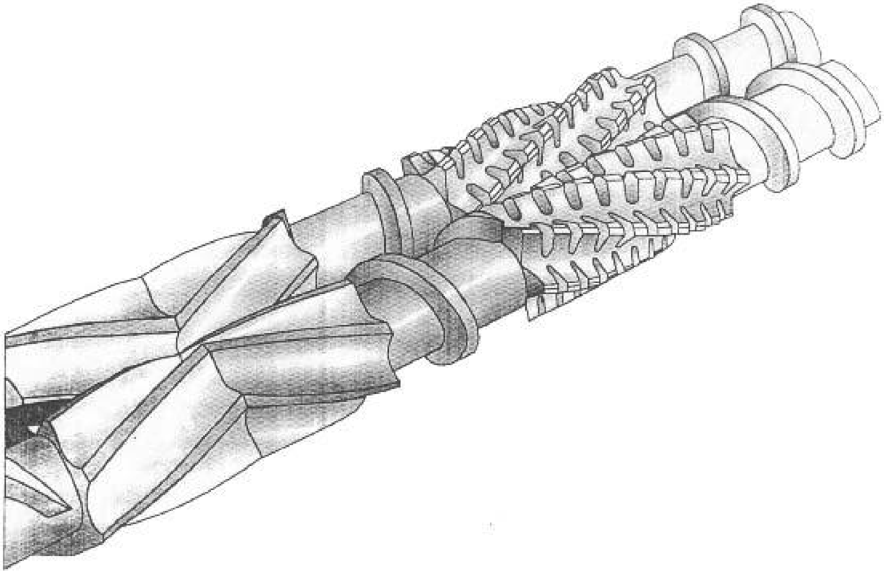


Figure 16-31 Hexalobal mixing screws.

The response to pressure generation is completely different, as backflow can easily occur through the gaps of the offset.

As with single-screw extruders [eqs. (16-4) to (16-6)], equations can be developed for drag and pressure flow terms for TSEs (Janssen, 1978; Todd, 1991). However, because of the great variety of screw and kneading paddle arrays, it is necessary to determine the relevant equations for each configuration in series.

It is also desirable to know the volume available for the various processing functions, such as reaction and devolatilization. For the common intermeshing bilobe configuration (Figure 16-33), the open cross-section (A) available for processing is

$$A = 3.08hD \tag{16-12}$$

where h is channel depth and D is screw diameter.

The fluid-conveying capacity, or drag flow Q_d, can be approximated by

$$\frac{Q_d}{N} = \frac{AZ}{2} \tag{16-13}$$

where N is the rotational speed and Z is the lead length (the axial distance required for 360° of a flight tip). Equation (16-19) is based on the observation that one-half of the material contained in one turn of the screw is conveyed forward per revolution. The degree of fill (f) in a barrel section where pressure

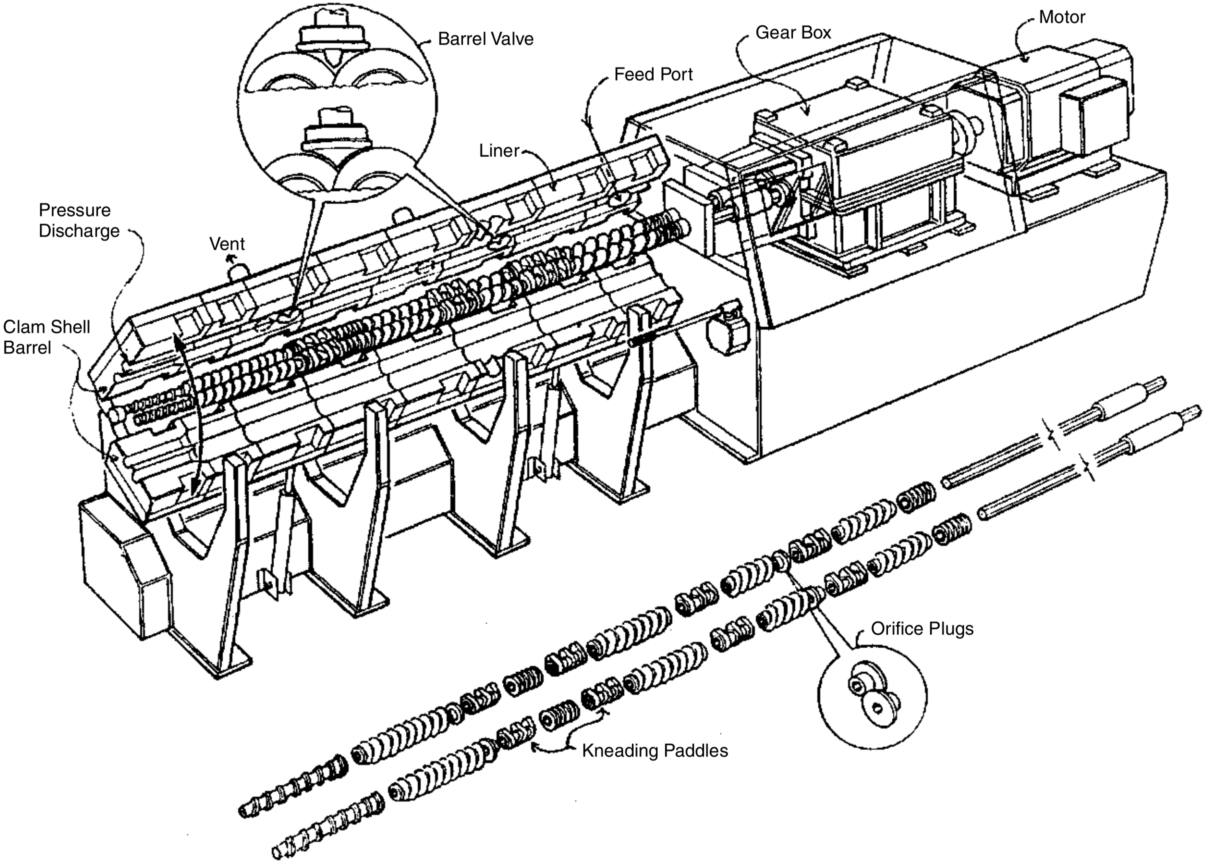


Figure 16-32 Co-rotating intermeshing twin-screw extruder with clamshell barrel. (Courtesy B & P Process Equipment and Systems, Saginaw, MI.)

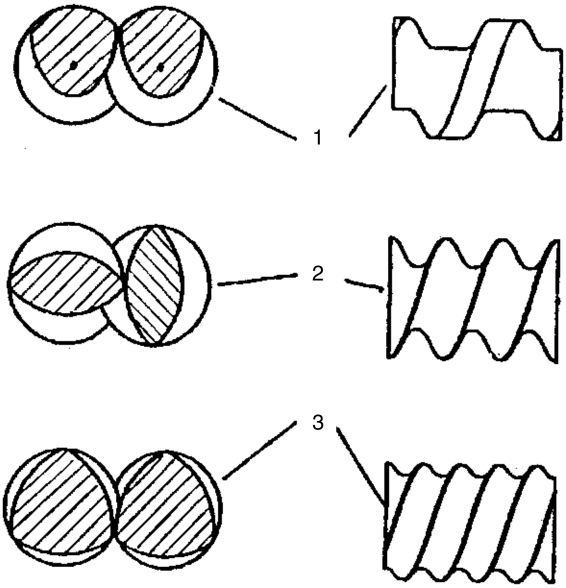


Figure 16-33 One-, two-, and three-start screw profiles for intermeshing co-rotating twin-screw extruders.

is not being generated is merely

$$f = \frac{Q}{Q_d} \tag{16-14}$$

where Q is the net flow.

The primary mixing action in intermeshing co-rotating TSEs is caused by multiple expansion/compression action as the rotors rotate. Figure 16-35 illustrates how the shaded material in one crescent section can receive material from upstream or downstream sections, and then be squeezed out a quarter-turn later. The elongational flow so produced is excellent for both dispersive and distributive mixing. This expansion/compression mixing action occurs only if that section of the barrel is completely full, so fill control is another necessary aspect of mixing in these TSEs (Todd, 1998). Additional mixing effects occur from the dispersive face of the kneading paddles (Figure 16-36) and the slicing action as the two rotors intermesh (Figure 16-37).

In addition to screw sections of differing helix angles, and kneading blocks of differing axial length, other special mixing elements are available, such as screws or blister rings with flight interruptions, as shown in Figure 16-38. The relative flow-pressure characteristics of such mixing elements have been reported by Brouwer et al. (2002). The screw devices are generally close to pressure neutral, as the slots in the flights are usually of opposite hand to the screw helix. The

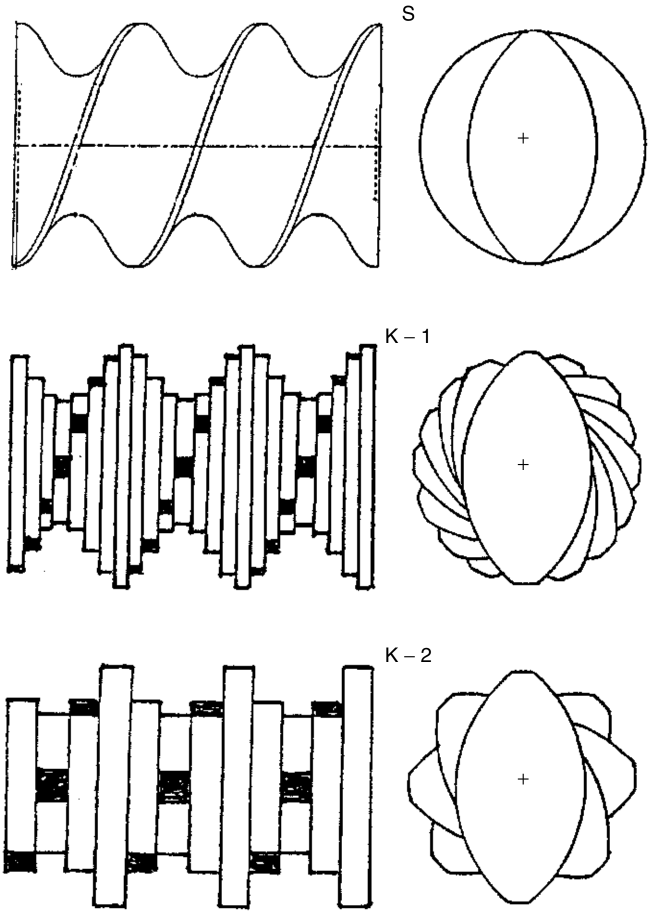


Figure 16-34 Kneading paddles in arrays matching square-pitch screws.

gear mixers (Figure 16-38, TME) may have neutral or angular slots either forwarding or reversing. These interrupted flight mixing elements provide multiple splitting and recombining actions. Residence time tests (Brouwer et al., 2002) indicate that these devices increase radial homogenization, with only a minimum of axial mixing. Hrymak and Wood (1999) have utilized a transparent acrylic TSE with the same index of refraction as their Newtonian test fluid to validate computational fluid dynamics models. The comparisons for the kneading disk region of an intermeshing co-rotating TSE were done using particle image velocimetry experimental data (Jaffer et al., 2000).

16-3.3 Special Mixers

Because viscous mixing needs are so varied, equipment manufacturers have created a broad range of devices to fill specific needs. Where a single agitator cannot

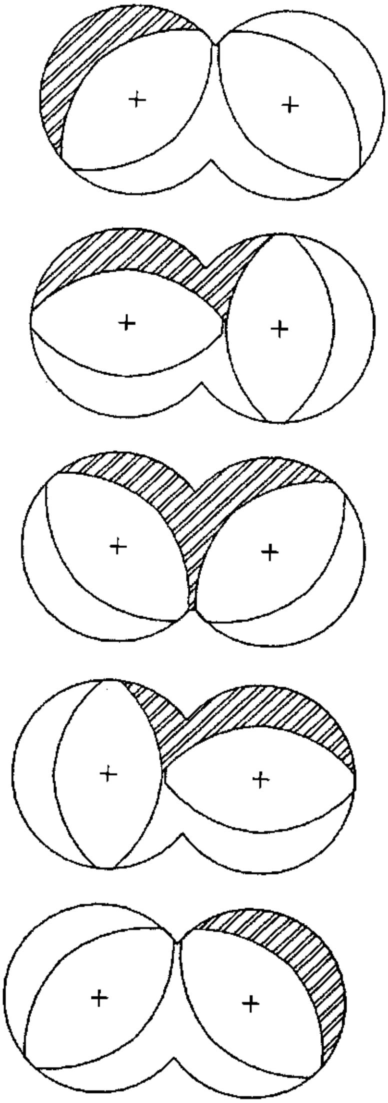


Figure 16-35 Expansion/compression mixing cycle in kneading paddles.

provide circulation and high shear simultaneously, a second mixing blade may be added. Blenders such as the plow mixer (Figure 16-18) may also be equipped with a high-speed chopper to break up clumps. Similarly, a high-speed impeller can be supplied along with an anchor mixer or with a combination anchor/helical blade agitator (Figure 16-39).

Pasty and tacky products frequently may be plagued with adherence to the agitator(s). Single-shaft mixers such as depicted in Figure 16-40, with additional

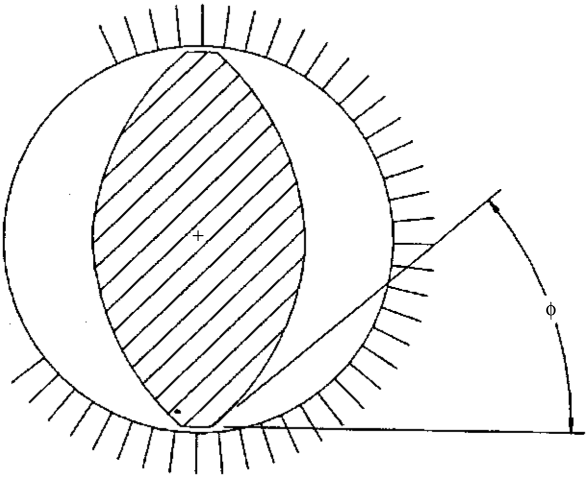


Figure 16-36 Dispersion face of a kneading paddle.

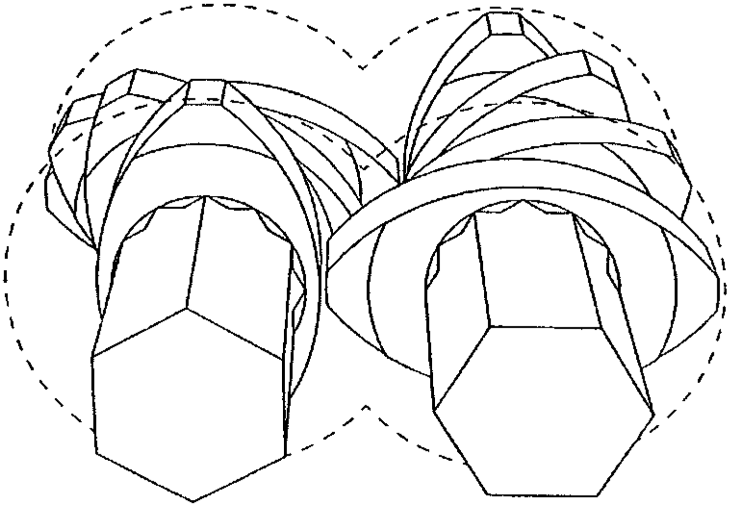


Figure 16-37 Slicing action between opposed pairs of kneading paddles.

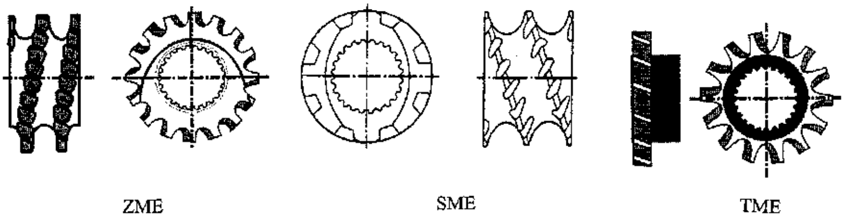


Figure 16-38 Mixing enhancers for intermeshing co-rotating twin-screw extruders.

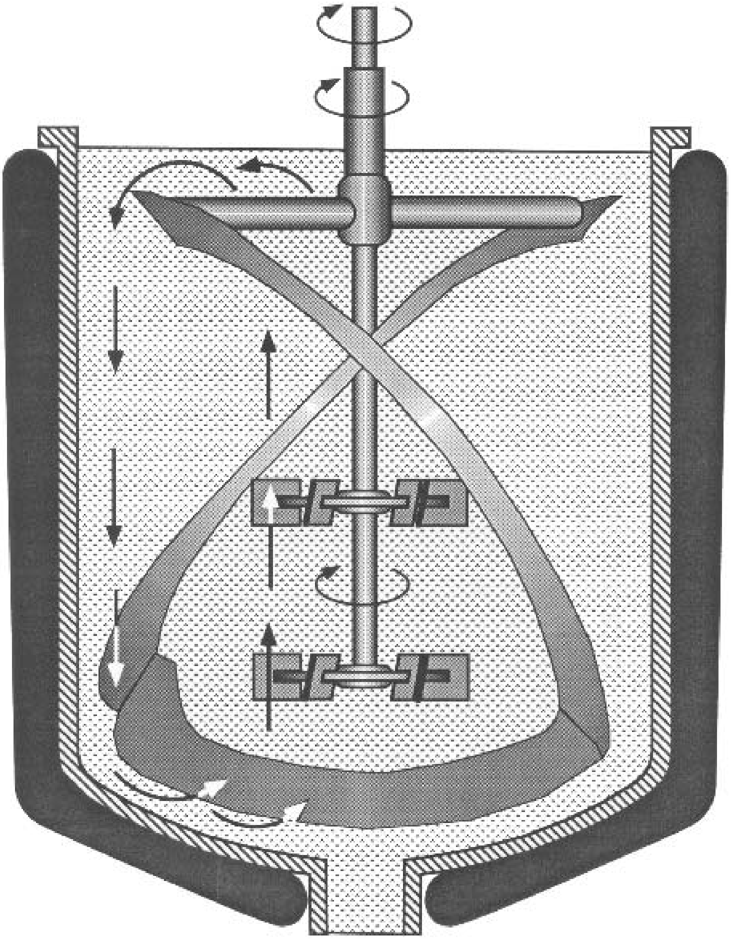


Figure 16-39 Combination anchor/helical blade, turbine mixer.

rotary scrapers for the disk blades, can operate in batch or continuous mode. Twin-shaft mixers (Figure 16-41) also are particularly suitable for mechanical and thermal processing with pasty, highly viscous, and crust-forming products.

16-4 EQUIPMENT SELECTION

The trade journals *Chemical Engineering* and *Chemical Processing* frequently run feature articles on mixing equipment. *Chemical Engineering's* annual *Buyers' Guide* provides a convenient list of mixer suppliers classified by type. *Plastics Technology's* annual *Processing Handbook & Buyers' Guide* has sections on extrusion systems and compounding and mixing systems. *Modern Plastics'*

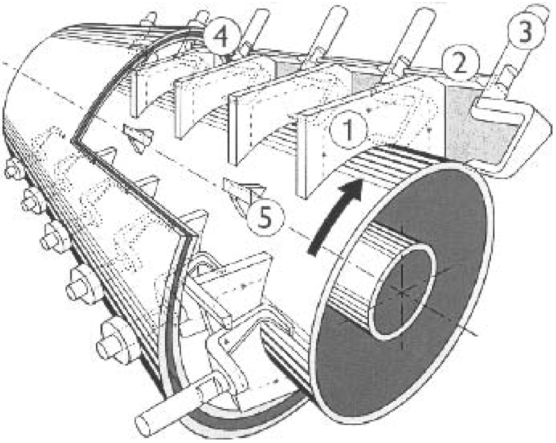


Figure 16-40 Batch thermal processor with wiped blades: 1, disk elements; 2, mixing/kneading bars; 3, counter hooks; 4, kneading space; 5, finger bars. (Courtesy of List, Inc., Acton, MA.)

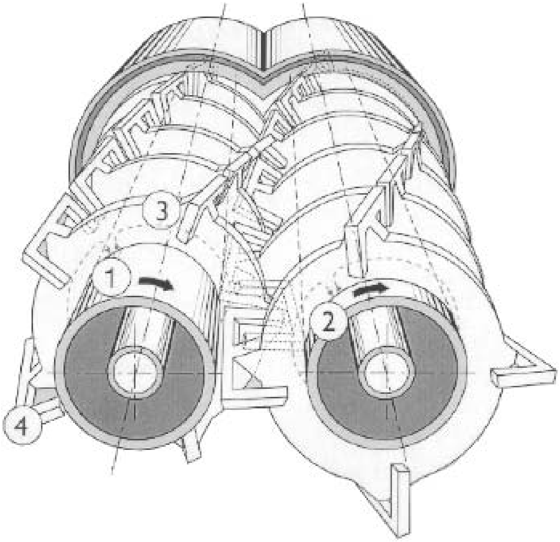


Figure 16-41 Twin-shaft continuous mechanical and thermal processor: 1, main agitator; 2, cleaning shaft; 3, disk; 4, kneading bars. (Courtesy of List, Inc., Acton, MA.)

annual *World Encyclopedia* includes a classified product listing of single- and twin-screw extruder suppliers, and descriptive text of extrusion, compounding, and mixing.

Most equipment vendors offer rental and pilot-size testing services and will take responsibility for sizing appropriate to meet the client's expected production-size requirements. During pilot testing, the sensitivity of the process to changes in shear rate, temperature, residence time, specific energy (kWh/kg), and any other special process requirement can be evaluated. Additional discussion is provided in Chapter 22.

With single-screw extruders, maintaining geometric similarity on scale-up usually does not provide an equal process result because of the importance of barrel surface temperature on the melting and mixing steps and the decrease in surface/volume ratio as diameter is increased. Scale-ups are usually limited to scale ratios of 2.5 : 1 or less.

Large twin-screw extruders operate almost adiabatically (typically, <10% of the energy imparted to the product can be transmitted through the barrel walls). Consequently, it is important to conduct pilot tests as close to adiabatic conditions as possible. Adiabatic operation permits scale-up of geometrically similar twin-screw extruders with capacity proportional to screw speed and the cube of diameter. Even though not much energy may be transferred through the barrel into the process, barrel temperature can still play an important role in controlling the temperature of the barrel wall film layer, and thus the viscosity and consequent viscous energy dissipation.

Specific energy (kWh/kg) is frequently a useful guide for achieving the desired mixed quality in the product (Irving and Saxton, 1967). Poor design, however, can lead to overmixing in one region and undermixing in another because of poor circulation.

Temperature control may be the most difficult task on scale-up. Putting more heat in is easy—you can always run the agitator(s) faster. Taking heat out is the problem. Consequently, one may have to run a large mixer at a lower rotational speed than the pilot unit, and thus it will take longer to reach the same level of homogeneity.

Because of the viscosity changes that may be taking place during the mixing process and the greater sensitivity of Reynolds number in viscous processing, it may be very difficult to model the system. Thus, it is better to rely on the experience of the equipment vendor for scale-up guidance.

16-5 SUMMARY

Mixing of viscous materials is the intermingling of two or more components by mechanical action of smearing, folding, stretching, wiping, and kneading. With the exception of static mixers (Chapter 7), where the fluid is forced around baffles in a pipe, the required state of product uniformity is achieved by rotation of one or more rotors within the processing vessel. Operation may be either batch or continuous. Viscous energy dissipation and poor heat transfer may limit the operating speed of the rotor(s) and thus prolong the time required when potential product degradation imposes an upper temperature constraint. Possible changes of state, and lack of information regarding the changing properties of the constituents during the mixing process, make a priori prediction of mixing performance of specific equipment most difficult. Equipment suppliers can provide rental equipment or conduct pilot tests to demonstrate feasibility and provide data required for scale-up.

NOMENCLATURE

a	dimensionless constant
A	area
b	dimensionless constant
c	dimensionless constant
C_p	heat capacity
D	diameter
e	flight width
E	screw pumping efficiency
F	shape factor
f	degree of fill
h	channel depth
h	heat transfer coefficient
k	thermal conductivity
K_p	proportionality constant
L	filled screw length
n	power law exponent
N	rotational speed
N_p	power number
Nu	Nusselt number
P	pressure
p	ratio of viscosity of dispersed phase to viscosity of continuous phase
Pr	Prandtl number
Q	volumetric flow rate
Re	Reynolds number
r	radius
T	temperature
t	clearance
W	channel width
Z	lead length of screw

Greek Symbols

α	dimensionless proportionality constant
γ	shear rate
μ	viscosity
ρ	density
σ	interfacial tension

Subscripts

c	continuous
d	dispersed
d	drag
L	large
p	pressure
S	small
w	wall

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