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MIXING OF PARTICULATE SOLIDS

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MIXING OF PARTICULATE SOLIDS

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MECHANISMS OF POWDER MIXING AND DEMIXING

Karl Sommer[x]

Classification of mixing process can be influenced
by significant motion in the mixers. While this
classification is only an empirical one, the clas-
sification in systematical or stochastical proces-
ses hints at internal mixing processes. In this
paper there is an special attention to the demi-
xing of powders. In principal there are two differ-
ent possibilities in demixing: 1. Demixing caused
by convective transport and 2. Demixing caused by
diffusion with concentration dependent diffusion
coefficient. The theoretical considerations give
hints for practical working. In order to study
these mechanisms, ballotini were mixed in a glass
drum.

INTRODUCTION

Mixing is one of the oldest unit operations. The earliest mixing
vessels that could positively be identified as such were mortars.
In ancient times, they were used in the production of natural
earth pigments such as ochre, manganese oxide, etc. Then, the mi-
xing process was accompanied by size reduction, and this feature
of dry-solids mixing has remained with us today. Despite the long
history of dry-solids mixing, or perhaps because of it, compara-
tively little is known of the mechanisms involved. All the experi-
ence that has been gained ever since solids were first mixed by
man some 30 000 to 40 000 years ago has been handed down through
the ages - formerly from medicine man to medicine man and nowadays
from foreman to foreman. By continuous trial and error, a degree
of perfection has been achieved that could hardly be improved upon
by scientific approaches.

Serious efforts at studying the dry-mixing of solids do not
extend back more than the last decade. In view of the growing num-
ber of tasks involving mixing techniques, the increasing demands
imposed on quality, and the trend towards rationalisation and me-
chanisation of processes, criteria must be found for comparing the
performance of various types of mixers. In other words, it is be-
coming more and more imperative to gain knowledge on the mecha-
nisms of mixing. The attendant problems can be divided into two
categories:

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a) Classifying and measuring the degree of mixing, i.e. determining the quality of the mixture (1 to 6).

b) Describing the mixing process, i.e. description and explanation of the change in the quality of the mixture with respect to time.

CLASSIFYING MIXING MECHANISMS ACCORDING TO THE PREDOMINANT FORM OF MOTION

Mixing mechanisms can be classified systematically according to the types of motion applied. An example for a classification of this nature has been put forward by Rumpf and Mueller (7).

a) Mixing within the bulk material

b) Centrifugal mixing α) in a gravitational field
                               β) in a centrifugal field

c) Mixing in a fluidized bed

d) Mixing solids in a suspended condition

e) Free-fall mixing

f) Mixing several streams of materials.

a) The first type of mixing consists of initiating within the bulk of the material a movement that displaces the solid particles relative to one another. The mixer or mixing elements must move slowly, an example being low-speed worm-type mixers. If the particles flow around the moving parts of the mixer, a state of stationary flow occurs within the bulk of the material.

b) In centrifugal mixing, some of the solids are detached from the bulk of the material and are rearranged in the air space of the mixer. They then return to the surface of the mixture. The force acting on the solids may be gravity or centrifugal force.

c) If mixing within the bulk of the material proceeds at high speeds, particles are thrown off, or air is drawn into the bed. Thus the particles move apart, and the bed assumes the character of a liquid. The fluidized state can be maintained by increasing the rate of air flow. This is the principle adopted in pneumatic mixers, which are gaining a firm foothold in practice.

d) Here, the solids are completely suspended in a gas. They can be kept in suspension by maintaining the flow of gas at a sufficiently high velocity or by agitation in high-speed mixers (8 - 11).

e and f) If the streams of solids to be mixed are metered and flow steadily, they mix in the desired proportions at the point of intersection. The solids may flow under gravity, e.g. in running out of a bunker, or they may be fluidized or conveyed pneumatically.

SYSTEMATIC MIXING

Ideally, the aim of any mixing mechanisms is to distribute the ingredients in such a manner that each particle of the one is adjacent to a particle of the other(s). In practice, the best that can usually be achieved is random distribution.

In the systematic processes that take place in all statistical mixers (12 - 14), streams of material are systematically distributed and rearranged and redistributed and rearranged ad infinitum.

Another form of systematic system is laminar mixing (Fig. 1), which is applicable to very viscous materials. It consists of introducing the material between two parallel plates that move relative to one another. At first, the particles of the ingredients are pulled wide apart, i.e. distributed, and they are then allowed to mix. A particular case is Couette flow in the annular gap beetween two concentric counter-rotating cylinders. Theoretically, any desired degree of mixing can be achieved by varying the number of revolutions.

In analogy to this laminar flow mixing model, mixing in layers can also be observed in solid mixtures (Fig. 2). However, the macroscopic physical laws pertaining to the bulk of the material are not so simple as those that apply to liquids. Thus, if the internal forces of friction acting on a crack that has been caused by fortuitous instability are overcome, the bulk of the material is loosened. This prevents the formation of a homogeneous field of shear in the gap and thus allows only a thin zone in which mixing can take place. If the gap is annular, mixing would occur only in a thin zone of shear, even after the mixer had rotated through an infinite number of revolutions; the material in the other zones would behave as a rigid unyielding body.

RANDOM MIXING

Random movements within the bulk of the material also improve the quality of the mixture. The feature of this form of mixing is that the particles are arranged at random and not in a predetermined manner throughout the mixture. Mueller and Rumpf (15) have demonstrated that random movements can be described, independently of the motion induced by the moving parts of the mixer, by the theory of stochastic processes (16).

Normally, more than one coordinate is required to fix the position of a particle. For the purposes of this paper, the systems are described in terms of the one single coordinate. In most practical cases involving mixers with purely axial movement, this is quite permissible if the indicator is initially inserted in a plane perpendicular to the axis or if mixing in the other directions is so rapid that the time that would be required is negligibly short compared to that required in axial mixing. If this is not the case, average concentrations can be taken perpendicular to the coordinate selected.

Mixing by diffusion. One of the probable random processes is diffusion. It can be described formally by Fick's equations (17).

$$\dot{M} = - D \frac{\partial q(x)}{\partial x} \qquad \text{Fick's first law} \quad (4.1)$$

$$\frac{\partial q(x)}{\partial t} = \frac{\partial}{\partial x} \left(D \cdot \frac{\partial q(x)}{\partial x} \right) \qquad \text{Fick's second law} \quad (4.2)$$

Fick's first law [Eqn. (4.1)] states that the mass flow $\dot{M}$ in a cross-section through the point x is negative proportional to the concentration gradient $\partial q(x)/\partial x$ at this point. The constant of proportionality D is referred to as the diffusion coefficient and is expressed in the unit of area/time. It is not a primary physical magnitude in the Fick partial differential equations but a fitting parameter that depends on the conditions of measurements. The diffusion coefficient might well be a function of concentration at point x. It is usually assumed to be positive for liquids; but, as will be demonstrated later, this is not necessarily the case for a stochastic process. If it is positive, mass flow is always in the direction of the lower concentration. Mixing always occurs if the diffusion coefficient is positive.

Fick's second equation [Eqn. (4.2)] describes the change in concentration at point x with respect to time. If the diffusion coefficient is positive and remains constant, a particular solution to the differential equation can be found for a simple drum mixer of length L (18). If it is assumed that the walls of the drum are impermeable, the limits can be obtained from the fact that there is no mass flow at x = O and x = L. In this case, the partial differential coefficient in Fick's first equation will become zero. The initial limiting condition can be satisfied by assuming that the ingredients are completely separated to start with. The solution thus obtained is a Fourier series (18). If the simplifying assumption is made that the one ingredient is present in very small amounts, the following cosinusoidal expression is obtained for $\Delta q(x,t)$, i.e. for the difference at time t between the concentration at point x to the average concentration $\bar{q}$ in the entire mixture.

$$\Delta q(x,t) = 2 \bar{q} \cos \pi \frac{x}{L} e^{- (\frac{\pi}{L})^2 DT} \qquad (4.3)$$

If the point x is fixed, Δq decreases exponentially with time. Thus if the coefficient of variation $\sigma' = \sigma/\bar{q}$ is taken as a measure for the quality of the mixture, the following simplified form is obtained:

$$\sigma' = \sqrt{2} \, e^{- (\frac{\pi}{L})^2 DT} \qquad (4.4)$$

On log-log paper, this relationship is a straight line, and the slope allows the diffusion coefficient to be determined experimentally. After infinite time, the concentration throughout the mixture will be entirely uniform, i.e. $\sigma' = 0$. However, the variance in the experiment is governed by sampling. Therefore, the measured value for the total variance contains an additional term that describes the random error in sampling (5, 18).

As an example, the experimental results obtained by Mueller (15, 18) have been plotted on a log-Iog scale in Fig. 3. In the experiment, a 300 - 400 μm copper fraction was mixed in a concentration of $\bar{q} = 7.67 \cdot 10^{-3}$ into a nickel fraction of the same size. A diffusion coefficient of $D = 1.09$ cm^2/sec is derived from the slope of the linear initial part of the relationship. It is almost independent of the length-to-diameter ratio. After a sufficiently long mixing time, the sampling error predominates, and the measured values depart from the straight line and asymptotically approach a fixed value. The final state can be interpreted as stochastically homogeneous to within the degree of accuracy allowed by the measurements.

Axial mixing with convective processes. If the ingredients in a mixture of powders have grains of different shape, size or density, demixing is frequently observed in stochastic mixing processes (19 - 27). It cannot be explained by the simple diffusion model with positive diffusion coefficients. For this purpose, Markhoff's fundamental theories on random processes must be resorted to. These theories eventually lead to the Kolmogoroff equations (16), which were introduced for the first time in statistical dynamics by Fokker (28) and Planck (29) (Fig. 4).

One imagines a large number of completely identical spheres that are independend with one another, e.g. ballotini in a matrix of large glass balls. Each of the spheres is in motion. At a certain time t, the concentration at a point x will be q(x). Each of the small beads is subjected to very light, rapid blows delivered at random. The question is how the concentration q(x) at the point x changes within a certain interval time τ. During this period of time, each of the spheres has received several blows, which may change the direction of motion. The sum of all the changes in location is expressed by the displacement Δz. Each sphere is displaced by a different amount Δz, even if they were all located at the same point x to begin with. The displacements Δz are statistically distributed with a probability density function of $\emptyset (\Delta z)$. Δz_{max} is the largest displacement that occurs in the period of time τ and must always be small compared to the dimensions of the mixer. $\bar{\Delta z}$ is the average displacement of the spheres, i.e. the mean convection. In addition to this convective movement, stochastic widening of the probability density function also occurs. It is a widening that is described by the mean square deviation $\bar{\Delta z^2}$, where

$$\overline{\Delta z^2} = \sigma^2 (\Delta z) + \overline{\Delta z}^2 \qquad (4.5)$$

Here, σ^2 (Δz) is the variance with the probability density function ϕ (Δz).

Since it is a square, $\overline{\Delta z^2}$ is always positive.

Fokker (28) and Planck (29) calculated the change in concentration $q(x)$ that occurs within the section dx from the difference between the number of particles that migrate from dx during the interval of time τ and the number of particles that enter dx during the same interval of time. The result was given in the form of a differential equation.

$$\frac{\partial q(x)}{\partial t} = - \frac{\partial}{\partial x} \left(q(x) \lim_{\tau \to 0} \frac{\overline{\Delta z}}{\tau} \right) + \frac{\partial^2}{\partial x^2} \left(q(x) \lim_{\tau \to 0} \frac{\overline{\Delta z^2}}{2\tau} \right)$$

(4.6)

As has already been stated, $\overline{\Delta z}$ represents the mean convection of small spheres at point x during the interval of time τ. Thus $\overline{\Delta z}/\tau$ is the mean rate of convection. If the limit $\lim \overline{\Delta z}/\tau$ exists, it is called the transport coefficient T and has the same unit as a velocity. If the limit $\lim \overline{\Delta z^2}/\tau$ exists, it is referred to as the dispersion coefficient D^+, and the unit is area/time, i.e. it has the same unit as the diffusion coefficient D. However, the dispersion coefficient is not a fitting parameter but is a statistically derived square and is thus always positive. Hence Eqn. (4.6) can be rewritten as

$$\frac{\partial q(x)}{\partial t} = - \frac{\partial}{\partial x} \left(q(x) \, T(x) \right) + \frac{\partial^2}{\partial x^2} \left(q(x) \, D^+(x) \right)$$

(4.7)

This equation has the same form as Fick's second equation, in common with which it describes the change in concentration with respect to time at the point x. The mass flow at a cross-section through the point x at time t is analogous to that in Fick's first equation, viz.,

$$\dot{M}(x) = q(x) \, T(x) - \frac{\partial}{\partial x} \left(q(x) \, D^+(x) \right)$$

(4.8)

Thus the mass flow consists of two components: transport by convection $\dot{M}_T$ and transport by dispersion $\dot{M}_D$. The simplifying assumption is made that $D^+(x) = D^+$, i.e. that D^+ is independent of the coordinate x and the transport coefficient $T(x)$ becomes zero, i.e. $T(x) = 0$. In this case, Eqn. (4.8) becomes converted into Fick's first equation (4.1), and the dispersion coefficient D^+ is identical to the diffusion coefficient. In a mixture whose ingredients are completely separated from one another at time t, equalization by diffusion takes place as described in Section 4.1. The variance σ_{tot}^2 in the sample concentration decreases steadily from the initial state σ_0^2 to the size of the sampling error σ_z^2 (Fig. 5 a).

If streams already exist for transporting the materials, two possibilities occur. If two streams are in the same direction,

their movement first of all favours equalization of concentration. Afterwards, however, the diffusion stream reverses and opposes transportation until both streams are of the same size. The stationary condition thus reached corresponds to a partially demixed state. Initially, the quality of the mixture rapidly improves but deteriorates again as demixing progresses (Fig. 5 b). If the two streams of material are in opposite directions from the very beginning, the concentration can never be completely equalized. The stationary state is partially demixed (Fig. 5 c).

The experiments carried out by Mueller (18) provide a good example of how these qualitative demixing phenomena can be demonstrated experimentally (Fig. 6). Demixing was bought about by the difference in transport characteristics between iron (630 - 750 μm) and quartz (100 - 200 μm) and between iron (300 - 400 μm) and limestone (40 - 60 μm). In the first experiment, the diffusion and transport streams were in the same direction; and in the second, in the opposite direction. The empirical coefficients of variation reflect the course of events predicted in the model. Hence, Experiment No. 1 first gave rise to a homogeneous mixture and then to demixing, and a random homogeneous distribution was not attained at any time in Experiment No. 2.

Axial mixing without convection processes. Demixing when the dispersion coefficient is constant, as was described in the previous section, always occurs if the moving parts in the mixer set up selective transportation that opposes mixing by diffusion. The same applies if the drum is tilted. Such demixing has also been observed to occur in drum mixers without the participation of external convection mechanisms. However, it has been demonstrated that if the transport coefficient tends to zero and the dispersion coefficient remains constant, the concentration is always equalized. For this reason, it should always be investigated whether the demixing observed can be explained by a dispersion coefficient that depends on the concentration or that is subject to local variations. Substituting $T(x) = 0$ in Eqn. (4.8) gives

$$\dot{M}(x) = - \frac{\partial}{\partial x} \left(q(x) \ D^+(x) \right) \tag{4.9}$$

If the product is first differentiated with respect to concentration and the concentration is differentiated with respect to x, an equation is obtained that is comparable to the first Fick equation.

$$\dot{M}(x) = - \frac{\partial \ (q \cdot D^+)}{\partial q} \ . \ \frac{\partial q(x)}{\partial x} \tag{4.10}$$

If it is assumed that the dispersion coefficient depends only on concentration, application of the product rule gives

$$\dot{M}(x) = - (D^+ + q \ \frac{\partial D^+}{\partial q}) \ \frac{\partial q(x)}{\partial x} \tag{4.11}$$

The term in parenthesis corresponds to the Fick diffusion coefficient, i.e.

$$D = (D^+ + q \frac{\partial D^+}{\partial q}) \tag{4.12}$$

Although the dispersion coefficient D^+ is, by definition, always positive, the diffusion coefficient may be negative. This is precisely the case when the relationship of D^+ to concentration gives rise to a sufficiently large negative differential coefficient. Under these circumstances, mass flow exists in the direction of higher concentration ; in other words, the ingredients demix.

In order to study these mechanisms, ballotini was mixed in a glass drum of 205 mm length and 114 mm diameter. First of all, pale and dark balls of the same size, viz. 2 mm diameter, were mixed in a ratio of 1 : 1. The mixer was 39 % full, and the speed was 45 r.p.m. Since the balls were of the same size and of the same material, it could be safely assumed that the dispersion coefficients were constant and independent of concentration. The contents of the drum were quickly mixed, i.e. within about 20 minutes, to stochastic homogeneity (Fig. 7).

If small glass spheres (of 1 mm diameter) are mixed with larger ones (of 2 mm diameter), the small balls will be more mobile in a matrix of larger balls than they would be if they were surrounded by balls of their own size. Thus the dispersion coefficient decreases with increase in concentration. If the decrease is significant enough, the diffusion coefficient may be negative. Under these circumstances, the ingredients, which were completely separated at first, would not mix. And, in actual fact, when practical tests were run, it was observed that the ingredients really did not mix, even after long periods of mixing. The contents of the drum remained demixed.

If there is a layer of small balls above the large ones, the concentration in the axial direction is completely equalized to begin with. For this reason, a diffusion stream cannot possibly exist. However, if it is assumed that the diffusion coefficient is negative, this equilibrium will be unstable. Disturbances can always occur because of the boundary conditions and of the irregularities in placing the balls in position. As a result, shifts in concentration could occur, and the possibility of demixing would arise. It can be seen that, in the tests (Fig. 8), disturbances of this nature occurred near the ends of the drum on the left-hand and right-hand sides. Within a mixing time of five minutes, the concentration of the smaller balls increased in the radial segments of these zones and decreased in the other zones. The demixing observed remained stable even during the periods of mixing. The number of disturbances depends on the length of the drum mixer. By varying the geometry of the drum, Donald and Roseman (19) were able to provoke numerous streaks.

The symmetry observed in this form cannot be expected unless the initial conditions are symmetrical. If disturbances are introduced in the form of local increases in concentration, individual streaks can be programmed at various points (Fig. 9).

On close observation, it can be recognized that the amount of small balls that have concentrated in streaks no longer accounts for half of the contents of the drum as it did at the beginning. Hence, many of the small balls must still be mixed in the matrix of large balls. This fact can also be explained by the theory. Eqn. (4.11) shows that the diffusion coefficient for a concentration $q \to O$ is always positive, even if the partial differential coefficient $\partial D^+/\partial q$ has a large negative value. Thus, at low concentrations, the ingredients can always be mixed. For instance, it could be assumed that there is a linear relationship between two dispersion coefficients for the small balls, i.e. D_1^+ at a concentration of $q \to O$ and D_2^+ at a concentration of $q = 1$. In this event, the limit for demixing in the first case (D_1^+) would be $q_{O1} = 0,5$. In the second case (D_2^+), it would be at the higher level of concentration q_{O2} (Fig. 10). If the concentration is lower than the limiting value q_O, the diffusion coefficient is positive, and the concentration will equalize by diffusion. If the concentration is higher than q_O, demixing results.

If the ratio of the dispersion coefficients is $D_1^+ : D_{23}^+ = 1 : 2$, the limiting concentration is at $q_{O3} = 1$. In other words, at this ratio and all others that are close to unity, demixing does not occur.

It was a mere assumption that the relationship to concentration was linear. Owing to the high limiting concentration of $q_O \geq 0,5$ for the system that was investigated experimentally, linearity could not be expected. However, if the relationship obeys, say, the Gauss error function, the demixing limit q_{OG} could lie at the point of inflection of the curve. Hence, it may occur at much lower concentrations, as is shown in Fig. 10.

Practical conclusions. The relationships postulated between the dispersion coefficient and the concentration are purely arbitrary. The first attempts to determine dispersion coefficients for solids originated from Cahn and Fuerstenau (30, 31), but unfortunately their results were not of general validity. Nevertheless, the method of considering mixing mechanisms as stochastic processes can lead to qualitative conclusions. Mueller (18) showed that, if there is a tendency to demix as a result of convection mechanisms, slight modifications, such as fitting or dismantling mixing elements, may alter the selective transport characteristic. Ullrich (23) recommended that tendencies towards convective demixing should be compensated, if possible, by judicious selection of the grain size and density.

If it is known that two ingredients tend to demix as a consequence of a negative diffusion coefficient, the dispersion coefficient must be made less dependent on the concentration. In practice, this is achieved by using fluidized bed and centrifugal mixers. By this measure, the mean free path between the particles becomes large compared with the size of the particles. Hence the mobility of an individual particle is less dependent on the size of those surrounding it. There is no doubt that the success of fluidized bed and centrifugal powder mixers can be attributed to the absolutely larger dispersion coefficient and the reduced dependence on concentration. However, they incur the great risk that the mixtures, although homogeneous at first, may become de-

mixed during the slow discharge and filling phases as a result of the dependence of the dispersion coefficient on concentration.

Another means of homogenizing materials that tend to demix is to add small amounts of a liquid (32). It has already been adopted in practice. The liquid forms bridges between the particles. Although the dispersion coefficients are greatly reduced as a consequence of the restricted mobility, the difference in the surroundings and thus the dependence on concentration no longer exert their dominating influence. As a result, a diffusive mixing process can be realized.

The addition of about 1 % wt of water to a charge consisting of 1 mm and 2 mm glass balls led to stochastic homogeneity within a mixing time of 10 minutes.

SUMMARY

Mixing mechanisms may be initiated by the predominant form of motion in a mixer, but this method of classification is purely empirical. Classification into systematic or stochastic processes gives an indication of internal mixing processes. Particular attention is drawn to demixing phenomena, which are of great significance for powder mixtures. A distinction can be made between two basic mechanisms – demixing caused by convection and demixing resulting from the dependence of the dispersion coefficient on the concentration. The theoretical considerations lead to qualitative practical conclusions.

LIST OF SYMBOLS

D = Diffusion coefficient (m^2/s)

D^+ = Dispersion coefficient (m^2/s)

L = Length of drum mixer (m)

$\dot{M}(x)$ = Mass flow at cross-section through the point x $(kg/(s \cdot m^2)$

$\dot{M}_D$ = Mass flow as a result of dispersion $(kg/(s \cdot m^2))$

$\dot{M}_T$ = Mass flow as a result of convection $(kg/(s \cdot m^2))$

$q(x)$ = Concentration at point x (kg/m^3)

$\bar{q}$ = Average concentration throughout the mixer (kg/m^3)

q_O = Limiting concentration between demixing and mixing (kg/m^3)

t = Time (s)

T = Transport coefficient (m/s)

x = Coordinate of particle in mixture (m)

Δz = Path through which a particle is displaced in axial mixing (m)

$\emptyset(\Delta z)$ = Probability density function for the displacement undergone by particles in axial mixing

σ^2 = Variance

σ = Standard deviation

$\sigma/\bar{q}$ = Coefficient of variation

τ = Interval of time in which the concentration q(x) changes at the point x (s)

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