Journal of Powder & Bulk Solids Technology, 2 (1978) 4; 2-8

MIXTURES AND MIXING OF MULTICOMPONENT SOLID PARTICLES – A Review

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INTRODUCTION

This review is focused on the literature related to the characterization of multicomponent solids mixtures and the mixing of multicomponent particle systems.

Solids mixing is an important unit operation in many industries. For example, it is essential in plastic processing, ore smelting, pharmaceutical preparation, fertilizer production, food manufacture, and catalytic synthesis of chemicals. This operation may be performed to achieve an acceptable product quality or to control rates of heat transfer, mass transfer or chemical reaction.

A convenient scheme for classifying particle systems should be established to facilitate the investigation of solids mixing. Particle systems may be classified mainly into two classes, binary and multicomponent systems. A binary particle system contains two different types of particles while a multicomponent particle system contains more than two different types. Strictly speaking, however, the binary particle system belongs to a special class of multicomponent particle systems.

Most of the early investigations on the mixture characterization and mixing were carried out with binary solids particle systems, which were identical in all aspects except for a small number of physical properties, e.g., color, which did not affect the mixing process. However, solid particles to be mixed often consist not only of particles of varying size and density but also of widely differing chemical and physical properties. Thus, the study of mixing and sampling of multicomponent heterogeneous solid particles is of practical significance.

Numerous publications on binary solids mixtures and mixing of binary systems are contained in a number of surveys and review articles, including those published by Weidenbaum[1], Bourne[2], Fan et al.[3-6], Valentin[7-9], Yano[10], William[11,12], Cooke et al.[13]. Since the study on the mixing of binary particle systems and their characterization is essential to the understanding of multicomponent systems, the recent publications on binary mixtures and mixing, which are not included in the previous survey and review articles, are also reviewed in this article.

There are two broad and equally important aspects in the study of solids mixing. One is concerned with the characterization of a mixture and the other with the mechanism and rate by which the state of a mixture changes. Both aspects are included in this review.

RECENT PUBLICATIONS ON BINARY SOLIDS MIXTURES AND MIXING

This section contains a brief review of the recent literature on mixtures and mixing of binary solids particle systems.

Mixtures

According to Schofield[14], three criteria are required to rigorously define mixture quality. These are between-sample variance (intensity of segregation), scale of segregation (small scale structure), and long range structure. Yip and Hersey[15] utilized Buslik's concept of homogeneity to define the homogeneity of a perfect mixture, which has zero standard deviation for the concentration of a minor ingredient between samples.

Eisenhart-Rothe and Peschl[16] introduced concepts of testing equipment for bulk materials handling and also described tests for characterizing the flow properties of powders. Wicks[17] blended solid particles with known particle size distributions to achieve a target particle size distribution. Akdel-Azim[18] developed a new technique for determining particle size of dispersing powders.

There are two approaches to the determination of the mixing index based on the contact number. One is based on the coordination number sampling, and the other on the spot sampling. By these two approaches, Shindo et al.[19,20] proposed theoretical models of the distribution of the contact number for a binary mixture in an incompletely mixed state.

Mixing

Rátkai[21] studied solids mixing in vertically vibrated beds. The effect of vertical vibration on the hydrodynamics of granular materials flow and the conditions necessary for the emergence of a regular particle stream were discussed. He also showed that the flow pattern in the bed and the change of velocities are functions of the vibration parameters.

Williams[11] reviewed three mechanisms of segregation: trajectory segregation, percolation of fine particles, and the rise of coarse particles on vibration. Koseki et al.[22] studied segregating phenomena in a horizontal rotating drum mixer. Steedman et al.[23] investigated solids segregation in a coal gasification burner. Sugimoto[24] studied solids mixing from some segregation characteristics of mixtures.

Segregation of powder particles by tapping or vibration, which is a phenomenon caused by a sifting of fines toward the bottom of the container, has been reported for the systems with particle diameters in the

NOMENCLATURE

$\underline{B}^{(i)}$	Sample amount proportion vector of the i-th sample
$\underline{B}$	Mean vector of $\underline{B}^{(i)}$
$C_{ij}(N)$	Number of concentration of component j in cell i at time Nt
$(C_j)_\infty$	Equilibrium concentration of component j
E_i	Corresponding expected number in the i-th sample, based on the average distribution of components in the mixture
$(\Sigma fw)_i$	Effective mean particle weight of component i
Hi	Negative logarithm of W_1 = $-\log W_1$
h_i	Number fraction of particles of component i in the mixture
I_s	Numerical index indicating degree of segregation of the mixture
k	Total number of components in the mixture
m(i)	Second moment about the origin of the volume distribution of particles of type i
m	$= \sum_{i=1}^{k} h_i\, m(i)$
N	Number of passes
n	Number of samples
O_i	Observed number of particles of any given color in the i-th sample
$P_{i\ell}$	Transition probability of the particles from cell ℓ
P_i	Weight proportion of component i
q_i	Theoretical volume concentration of component i
q_j'	As defined in equation 21 $= \dfrac{q_j}{1 - \sum_{i=1}^{j-1} q_i}$
Q_{ij}	As defined in equation 11 $= \Pi_i \dfrac{C_{ij}(0)}{(C_j)_\infty}$
r	Number of revolutions of the mixer
$\underline{S}$	Sample variance-covariance matrix
T	Testing statistic as defined in equation 6
t	Required time for one pass
V	Volume of particles in each sample
v_i	Volume of a particle of component i
v	Average volume of a particle in the mixture
W	Weight of a sample
W_1	Weight of sample necessary to give a standard deviation of 1%
w	Effective mean weight of all particles in the mixture
X_i	Volume concentration in a sample

Greek

α	Total number of cells in a mixer
η	Rate constant
λ	Fixed total amount of a sample
$\mu(i)$	Mean of the size (or volume) distribution of particles of type i
μ	As defined in equation 5 $= \sum_{i=1}^{k} h_i \mu(i)$
Π_i	Number fraction of particles in cell i
Σ_r	Variance-covariance matrix of a mixture in the completely mixed state
Σ_s	Variance-covariance matrix of a mixture in the completely segregated state
σ_i	Standard deviation of volume fraction of size v_i in random samples of constant volume V
χ_n^2	chi-square value with n degrees of freedom
χ_o^2	Observed chi-square value for a mixture
χ_r^2	Expected chi-square value for a random mixture
χ_s^2	Expected chi-square value for a segregated mixture

range of 0.2 to 12 mm[25]. Parson[26] extended this effort to micrometer-sized particle systems with and without the presence of agglomerates.

Rowe and Nienow[27] reviewed critically some of their earlier works on particle mixing and segregation in gas fluidized beds. They also reported briefly on recent works and drew attention to present unsolved problems. Burgess et al.[28] used gas fluidized beds to study solids mixing and segregation. They found that mixing commences at gas velocities just above the minimum fluidization velocity of the non-segregating component for particle systems of equal density, but mixing does not commence until the gas velocity exceeds the minimum fluidization velocity of the segregating component for systems of different density. They also developed a model for segregation and mixing, which is capable of predicting the complex behavior found experimentally.

Fitzgerald et al.[29] used a fluidized bed to study solids mixing and calculated solids flow patterns by use of a general compartmental model. Rengarajan et al.[30] employed the single phase backflow cell model to simulate the solids mixing in a fluidized bed coal combustor.

Highley and Merrick[31] developed a mathematical model to calculated the lateral concentration gradients of reactant solids in a large bed and used it to predict the effect of coal feed spacing on the combustion of coal in a fluidized bed boiler. Lateral-solids mixing diffusivities were measured in a 5 ft. diameter bed.

The economic advantages of using a continuous process are now widely accepted. However, processes involving the handling and processing of particulate solids are often designed as batch processes. Such systems should be examined to determine if it would be advantageous to employ continuous mixing. Williams[12] reviewed the continuous mixing of solids, in which the advantages of continuous mixing and the equipment for continuous mixing are also reported. Miyanami et al.[32] developed a continuous mixing process for fine and cohesive powders. The vertical cylinder mixer provided with an impeller of multistage comblike blades has been proven effective. Carstensen[33] showed that the mixing of a binary mixture of non-spherical particles with rough surfaces obeys to diffusional equations as long as the mean diameters of the two fractions are identical. Hogg and Hwang[34] carried out experiments on the mixing of a thin layer of tracer fed onto the surface of a flowing bed of sand. The results of their experiments were analyzed in terms of a diffusion model in which it is assumed that the diffusion coefficient is a function of the velocity gradient at any level in a flowing stream.

Wang and Fan[35] presented discrete deterministic and random walk models to describe the axial mixing of grains in a motionless Sulzer (Koch) mixer. They[36] also proposed a discrete steady-state Markov chain model for the axial segregation of solid particles in a motionless mixer. This model can predict the concentration profiles, the degree of mixedness, and the equilibrium states of these particle mixtures blended by passing the particles through a motionless mixer.

Thýn and Duffek[37] investigated the mixing process in a horizontal batch mixer with a twin spiral rotor. Krambrock[38] used a pneumatic mixer unit to study the mixing and homogenizing of granular bulk materials. Bridgwater[39] reviewed fundamental powder mixing mechanisms. Yamaguchi[40] developed some rate equations for solids mixing.

MULTICOMPONENT MIXTURES AND MIXING

The publications on multicomponent mixtures and mixing are reviewed extensively in this section.

Mixtures

Gayle et al.[41] used chi-square as a criterion to characterize the homogeneity of a multicomponent solids mixture instead of a conventional mixing index based on the sample variance (or standard deviation). According to them,

$$\chi^2_n = \sum_{i=1}^{n} \frac{(O_i - E_i)^2}{E_i} \quad (1)$$

where

n = number of samples,
χ^2_n = chi-square with n degrees of freedom,
O_i = observed number of particles of any given colour in the i-th sample,
E_i = corresponding expected number in the i-th sample, based on the average distribution of components in the mixture.

They also proposed a segregation index as

$$I^2_s = \frac{\chi^2_o - \chi^2_r}{\chi^2_s - \chi^2_r} \quad (2)$$

where

I_s = numerical index which indicates degree of segregation of the mixture,
χ^2_o = observed chi-square for any mixture,
χ^2_r = expected chi-square for the random mixture,
χ^2_s = expected chi-square for the segregated mixture.

Buslik[42] developed a formula for the variance of a single size fraction of the given particle size v_i in random samples from a granular material with a known size distribution as

$$\sigma_i^2 = \frac{q_i (1 - q_i) v_i^2 + q_i^2 (v - v_i)}{V} \quad (3)$$

where

V = volume of solid material in each sample,
$\bar{v}_i$ = average volume per piece of size v_i being investigated,
q_i = volume fraction of an aggregate which has size v_i,
σ_i = standard deviation of volume fraction of size v_i in random samples of constant volume V and
$\bar{v}$ = average volume of a particle in the mixture

v is defined as

$$v = q_1 v_1 + q_2 v_2 + \ldots + q_k v_k$$

where $q_1, q_2, \ldots, q_k$ are the volume fractions of the particle sizes $v_1, v_2, \ldots, v_k$ in the aggregate.

Stange[43] characterized the quality of a mixture consisting of granular particles of different sizes, denoted by 1,2,3,... He developed the following formula for the variance of one of the components by lumping the remaining components as a fictitious component.

$$\sigma_a^2 = \frac{p_1}{W}\left[\left(\frac{1-p_1}{p_1}\right) p_1 (\Sigma f_j w_j)_1 + p_2 (\Sigma f_j w_j)_2 + p_3 (\Sigma f_j w_j)_3 + \text{etc}\right] \quad (4)$$

where

p_1, p_2 etc. = weight portions of component 1, 2, etc.,
$(\Sigma f_j w_j)_i$ = effective mean particle weight of component i.

This expression can be confirmed by extending Buslik's equation (3) to cover multicomponent system[44].

Knott[45] analysed a mixture of particles of different sizes by employing a technique used in the study of a joint number of occurrences of various types in a renewal process up to a fixed time. He derived expressions for the asymptotic means, variances, and covariances of the volumes of different types of particles as the total volume sampled becomes large. The obtained asymptotic variance of type i, σ_i^2 is

$$\sigma_i^2 = \frac{Vh_i\mu^2(i)}{\mu^3}\,[\mu - h_i\mu(i)]^2 + h_i\,[m - h_i\mu^2(i)] + \frac{Vh_i\,[m(i) - \mu^2(i)]}{\mu^2}\,[\mu - 2h_i\mu(i)] \quad (5)$$

where

h_i = number fraction of particles of type i in the mixture,
V = volume of particles in each sample,
$\mu(i)$ = mean of the size (or volume) distribution of particles of type i,
$m(i)$ = second moment about the origin of the volume distribution of particles type i.

$$\mu = \sum_{i=1}^{k} h_i\,\mu(i)$$

$$m = \sum_{i=1}^{k} h_i\,m(i)$$

k = number of types of different particles

Scheaffer[46] applied the renewal theory to find the asymptotic distribution of the sample amounts of the different types of items, to estimate the population amount proportions, and to estimate the average item amount. His results of asymptotic variance and covariance are essentially the same as Knott's[45]. Scheaffer[47] proposed a homogeneity index for a multicomponent mixture. He assumed that the population mean and covariance are known. A test statistic, T, was proposed as

$$T = \lambda \sum_{i=1}^{n} (\underline{\hat{B}}^{(i)} - \underline{B})'\,\Sigma^{-1}(\underline{\hat{B}}^{(i)} - \underline{B}) \quad (6)$$

where λ is the fixed total amount of a sample, n is the number of samples, $\underline{\hat{B}}^{(i)}$ is asymptotically distributed as a multivariate normal random vector with the mean vector, $\underline{B}$, and a covariance matrix, $\lambda^{-1}\Sigma$. From the asymptotic normality of $\underline{\hat{B}}^{(i)}$, T has a $\chi^2_{n(k-1)}$ distribution as $\lambda \to \infty$ asymptotically under the hypothesis of a thoroughly mixed aggregate. T should be close to the mean value of this χ^2 distribution for a thoroughly mixed aggregate, and is much larger for a poorly mixed one. But this test statistic does not give a good approximation of the exact distribution except in the case of a very large sample.

Buslik[48] introduced a concept of homogeneity of binary mixtures based on the weight of sample, W_1, necessary to give a specified variation (e.g., a standard deviation of 1%) between samples. The so-called universal homogeneity is defined as the negative logarithm of the sample weight. Hersey et al.[49,50] extended this concept to a multicomponent mixture by using the standard deviation of an ingredient 1 in a completely random mixture, (equation 4). They assumed that the components must be reduced to the same particle-size level prior to mixing, i.e., particles of each component have the same particle weight.

$$\Sigma(f_j w_j)_1 = \Sigma(f_j w_j)_2 = \cdots = w \quad (7)$$

where w is the effective mean weight of all particles in the mixture. Thus, equation (4) reduces to

$$\sigma_1^2 = \frac{w}{W} p_1 (1 - p_1) \quad (8)$$

When $\sigma_1 = 0.01$ and $W = W_1$ gives a different equation for the homogeneity of an individual ingredient in the multicomponent mixture as

$$Hi(j) = -\log\ p_j(100 - p_j)w\ ,\ j = 1,2,\ldots \quad (9)$$

The equations can be used to follow the course of mixing of a single component operation or to compare the various requirements of powders necessary for mixing them to a desired degree of homogeneity in a multicomponent mixing operation.

Cook and Hersey[51,52] evaluated a Nauta DX 600 mixer for the mixing of a multicomponent tablet preblend. Four ingredients (phenobarbitone, butobarbitone, quinalbarbitone and lactose) were mixed. During the mixing operation, samples were removed from the mixer and assayed for each ingredient by using gas liquid chromatography. The degree of mixing for each of the four components was calculated according to equation (9). Each component behaved in a unique manner during mixing. The control of mixing time is very important for multicomponent mixtures, since at any one time one ingredient may have segregated to the extent that it does not meet the necessary specification for homogeneity.

Lai and Fan[53] proposed a discrete steady-state Markov chain model for the mixing process of a multicomponent homogeneous particle system in a motionless mixer. The concentration variance, σ_N^2, of the mixture at time Nt is given by

$$\sigma_N^2 = \frac{1}{k} \sum_{j=1}^{k} \sum_{i=1}^{\alpha} \Pi_i\,[C_{ij}(N) - (C_j)_\infty]^2 \quad (10)$$

where

$C_{ij}(N)$ = number concentration of component j in cell i at time Nt,
$(C_j)_\infty$ = equilibrium concentration of component j,
Π_i = number fraction of particles in cell i,
α = total number of cells in a mixer,
k = total number of components in a mixer.

The variance of sample compositions in the completely segregated state, σ_o^2, was derived from the model as

$$\sigma_o^2 = \frac{1}{k}\sum_{j=1}^{k} \Pi_i [C_{ij}(0) - (C_j)_\infty]^2$$
$$= \frac{1}{k}\sum_{j=1}^{k}\sum_{i=1}^{\alpha} [(C_j)_\infty]^2 \Pi_i \left[\frac{1}{\Pi_i}\sum_{\ell=1}^{\alpha} P_{i\ell}(N)\, Q_{i\ell}(0) - 1\right]^2 \quad (11)$$

where

$P_{i\ell}$ = transition probability of the particles from cell i to cell ℓ,

$$Q_{ij}(0) = \Pi_i \frac{C_{ij}(0)}{(C_j)_\infty}$$

The variance reduction ratio, Ψ, can be written as

$$\Psi = \frac{\sigma_N^2}{\sigma_o^2} \quad (12)$$

The degree of mixedness may be defined as

$$M = 1 - \Psi \quad (13)$$

Lai and Fan[53] proposed the idea of using the entropy of a solid mixture as a criterion for characterizing the mixture. The entropy of the solid mixture at cell i can be defined as

$$S_i(N) = -\sum_{j=1}^{k} C_{ij}(N)\ln C_{ij}(N) \quad (14)$$

and the total entropy of the system is then

$$S(N) = \sum_{i=1}^{\alpha} \Pi_i S_i(N) \quad (15)$$

If the system is in the segregated state at the beginning of the mixing process,

$$S_i(0) = 0 \quad (16)$$

and therefore,

$$S(0) = \sum_{i=1}^{\alpha} S_i = 0 \quad (17)$$

The entropy of a mixture will change from zero in the completely segregated state to a constant value in the completely random state. Hence, the entropy of a mixture can be similarly defined as a measure of the degree of mixedness of a mixture.

Wang et al.[54] applied multivariate statistical methods to test a variety of hypotheses which consist of the equality of mean vectors and that of covariance matrices. The applicability of the multivariate statistics to analyses of mixing processes and mixtures of multicomponent particles has been successfully demonstrated. They have defined the degree of mixedness, M, for a multicomponent solids mixture as

$$M = \frac{|\Sigma_s| - |S|}{|\Sigma_s| - |\Sigma_r|} \quad (18)$$

where

$|S|$ = determinant of the sample covariance matrix,
$|\Sigma_s|$ = determinant of the covariance matrix in the completely segregated state,
$|\Sigma_r|$ = determinant of the covariance matrix in the completely random state.

M assumes the values of O and 1 in the completely segregated state and the completely random state, respectively.

In mixing several monosized particle fractions, Sommer[55] assumed that the volume of a particle of a given size, v_i, is always an integral multiple of that of the next smaller size particle, v_{i+1}, i.e.,

$$v_1 = \mu_2 v_2 = \mu_2\mu_3 v_3 = \ldots, \quad \mu_i = 1, 2, \ldots \quad (19)$$

where v_1 is the volume of the particle with the greatest size and v_n the smallest size. Under this assumption, the formula can be used to calculate the variance of the stochastic homogeneity as

$$\sigma_r^2(X_j) = q_j(1 - q_j')\frac{v_j}{V_p} + q_j'\sigma_r^2\left(\sum_{i=1}^{j-1} X_i\right) \quad (20)$$

and

$$\sigma_r^2\left(\sum_{i=1}^{j} X_i\right) = q_j(1 - q_j')\frac{v_j}{V_p} + (1 - q_j')\sigma_r^2\left(\sum_{i=1}^{j-1} X_i\right) \quad (21)$$

where

$$q_j' = \frac{q_j}{1 - \sum_{i=1}^{j-1} q_i},$$

X_i = volume concentration in a sample,
q_i = theoretic volume concentration of component i.

Mixing

Gayle et al.[41] carried out mixing experiments by using a mixing wheel, which was designed for mixing samples of coal and coke. Granules which were similar in surface characteristics, shape and density but different in colour, were used. During mixing, the value of chi-square, as defined in equation (2), decreases with an increase in the number of revolutions of the mixing wheel and approaches a mean lower limit asymptotically. The initial value, χ_s^2, for chi-square (before mixing) is equal to the number of degrees of freedom multiplied by the number of particles counted for each sample. The number of degrees of freedom is defined as the number of items of data. The segregation index of equation (3), which varies from unity to zero as mixing proceeds from complete segregation to complete randomization, was used to establish a conventional rate equation as

$$-\frac{dI_s}{dr} = \eta I_s^{\theta} \tag{22}$$

where

I_s = degree of segregation
r = number of revolutions of the mixer,
η = rate constant,
θ = constant

Lai and Fan [53] developed a stochastic model for the mixing of multicomponent homogeneous particles by passing them through a motionless mixer from the experimentally determined transition probabilities of a binary homogeneous particle system. The model permits prediction of the concentration distribution and the degree of mixedness of a multicomponent homogeneous particle mixture.

Wang et al. [54] employed a drum mixer to study multicomponent solids mixing. Three types of particles having identical properties except color were used. They indicated that the determinant of a sample covariance matrix, $|S|$, monotonically decreases as the mixing time increases. They also showed the mixing index M, as defined in equation (18), increases and approaches 1 as the mixing time increases. For mixing a multicomponent heterogeneous mixture, it is expected that M increases up to a certain maximum and decreases asymptotically to the equilibrium state because of the segregating tendency of such a mixture.

CONCLUDING REMARKS

This review of the literature on binary mixtures and mixing indicates that mixing of binary components systems in fluidized beds have been fairly extensively studied in the last several years. This is a natural consequence of the fact that the use of fluidized beds in many areas of material processing is increasing rapidly. This review also indicates that segregation or demixing, which is the counterpart of mixing, rather than mixing itself has become the emphasis of the recent investigations.

Mixing and sampling of multicomponent heterogeneous solids mixtures and their interrelations have been of intense interest. Yet, the literature on this subject is still very limited. For instance, the criteria for testing the homogeneity of a heterogeneous multicomponent mixture is still lacking. Since any knowledge of multicomponent solids mixing and mixtures should reduce to that of binary mixing, to establish a broad, systematic and analytic approach to this subject is of importance and significance.

ACKNOWLEDGEMENT

The authors wish to acknowledge the financial support of the National Science Foundation (Grant ENG 73-04008A02).

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