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THESE

présentée à l'Ecole des Mines d'Albi-Carmaux

pour obtenir

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MODELES MARKOVIENS POUR LE MELANGE DES POUDRES EN MELANGEUR STATIQUE

Soutenance prèvu le 09/11/2006

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NOMENCLATURE

b - transition probability of material going up;

c - concentration;

CV - coefficient of variation;

D - diffusion coefficient;

d - particle diameter [m];

d₁₀ – accepted minimal diameter of particles [m];

d₅₀ – average diameter of particles [m];

d₉₀ - accepted maximal diameter of particles [m];

k - the number of mixer revolutions;

L – the number of sections;

m – the number of mixer cells;

 $\mathbf{M}_{A,B}$ – the matrices of transition probabilities for the components A and B respectively;

m_{ph} – mass of mixture after one cycle (a cycle here is considered to be the process time from charging to discharging) [kg];

m_{req} – required mass of final product [kg];

n - the number of samples taken;

N – the number of sections in the mixing zone;

P - the matrix of transition probabilities;

pa-transition probability to the absorbing state;

 p_b – transition probability from the further cell to the previous cell;

p_c – transition probability in the horizontal direction;

p_{cA} - probability of the component A to transit horizontally (2D model);

 p_{cB} - probability of the component B to transit horizontally (2D model);

 p_d – transition probability in vertical direction of the mixing zone;

p_f – transition probability to the further cell;

 p_{ij} – transition probability from j-th to i-th state;

p_s – transition probability to remain in a cell after one transition;

 p_{sA} – probability of the component A to stay in a cell (2D model);

 p_{sB} - probability of the component B to stay in a cell (2D model);

 $p_{A,} p_{B}$ – transition probabilities to a further cell for the components A and B respectively;

 $q_e(x,t)$ – function of source density;

S - mean standard deviation;

S - state vector;

S $_{i}$ – elements of the state vector;

 S_1^0 – initial state vector;

 S_{z}^{0} - state vector of material feed into the mixer;

S_A, S_B – state vectors for the components A and B respectively;

 S_{fA1} , S_{fB1} – state vectors of material feed into the first cell of the mixer the components A and B respectively;

 $S_{openflow}$ – is the open flow area [m²];

SPAN - relative diversity of particle sizes with regard to d₅₀;

 S_{za} , S_{zb} – state column vectors for material in the columns of the loading container for the components A and B respectively;

t – time;

t1 - time of particle motion along the loading container till the first row of the screws [s];

t₂ - time of particle motion through the mixing zone [s];

t_{cycle} – total cycle time [s];

thandling - handling time [s];

t_{loading/unloading} - the time of mixer loading and unloading [s];

t_{passage} – time of one passage [s];

u - transport coefficient;

VRR – variance reduction ratio;

X – state space;

X_n – stochastic variable;

z - number of transition;

Greek letters

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\sigma_m^2 – variance;
```

 $\alpha_1, \alpha_2... \alpha_n$ – delay coefficients;

 μ – average concentration;

 ρ_s - particle density [kg/m³];

 $\upsilon-\text{transport coefficient;}$

 ϵ – mixer porosity;

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1 GENERAL INTRODUCTION

<u>Motivation and background.</u> New technologies of chemical process industry are extending the usage of particulate solids in the mixture state. Mixing of powders, particles, flakes, fibers, granules gains increasingly economical importance in different types of industry ranging from mixing of human and animal foodstuff, pharmaceutical products, detergents, chemicals, plastics, etc. In most cases, mixing process adds significant value to the product and it can be regarded as a key process. By mixing, a new or intermediate product is created. The quality and the price of this product often depend on mixing efficiency. Both design and operation of the mixing unit itself have a strong influence on the quality produced. Upstream process steps like feeding, sifting weighing, transport etc. also determine quality of the process.

The term mixing is applied to operations which tend to reduce nonuniformities or gradients in composition, properties, or temperature of material in bulk. Such mixing is accomplished by movement of material between various parts of the whole mass [3].

By far, the most important use of mixing is production of homogenous blend of several ingredients that neutralizes difference in concentration inside the volume of a batch. Continuous mixing process also neutralizes inflow fluctuations of the components.

The technical process of mixing is performed by a multitude of equipment available on the market. However, mixing processes are not always designed with the appropriate care. This causes a significant financial loss which arises in two ways:

a) <u>Mixture quality is poor</u>, it will be noticed immediately at the product quality inspection. However, mixing is often one of further processing stages. Here the effects of unsatisfactory blending are less apparent. b) <u>The homogeneity is satisfactory but too much energy consumed for achiev-</u> <u>ing it</u>. As a result, segregation may appear within the scale of scrutiny while mixing components with different physical properties. If to mix some sensitive products, long mixing time can cause their deformation. If the mixing process is not optimally configured, many numerous pieces of equipment might be used than it would be necessary what increases the production cost.

While mixing is analyzed, the following three questions should be answered:

- how good mixing is;
- how quickly the mixture state will be reached;
- how high the required energy input is.

However, research on these problems with analyzing different materials and conditions for achieving necessary quality by means of direct empirical work is a long and expensive procedure. That is why the importance of mathematical models is growing up. The models can reduce dramatically the empirical work required for predicting mixture parameters. It results from the fact that a couple of simple experiments can be made to determine parameters of a model and afterwards the model will give us information about the process when mixing conditions change. Without the model, it would require many experiments with the equipment to be able to find the best regime or to investigate how this equipment reacts to the changes of mixing conditions.

Thus, simulation results in cutting costs of empirical work and decreasing the time for experiments. However, the models built for this purpose, usually describe a certain type of mixers, what does not make these models the general strategy of mixing process simulation. Besides, the majority of such models does not allow direct experimental identification of their parameters which could be found on the basis of independent experiments. This circumstance does not make it possible to reduce the experimental information with keeping reliability on prediction of mixture parameters.

Static mixers with multiple passages of material through the mixing zone are a very interesting subject of research and mathematical simulation. They have a position between continuous mixers and batch mixers in a closed volume so that static mixers combine advantages of both operation principle types. The mixers bring a good deal of technological interest while mixing of segregating materials because while rotating the mixer, components change their place. It is possible to search for the number of material passages through the mixing zone giving better mixture quality, and to stop the process if the necessary quality is achieved. Absence of rotating

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parts inside the mixing zone improves their technological reliability. Although batch loading reduces the throughput, it significantly improves the feeding precision. However, research on these mixers is oriented on the direct experimental investigation of the mixture quality [36, 37, 46-48, 51], but not the conditions of obtaining such quality. Thus, it is more essential to build models that can predict efficiency of mixer operation in the conditions changed. Therefore, further theoretical and experimental research of mixing in static mixers is a representative scientific and technological problem. It results in the objective of this study which is carried out within the program "FTSP, Integration" (2.1 – A118 Mathematical modeling resource-saving and ecologically safe technology) and the agreements on scientific cooperation between Ivanovo State Power Engineering University, Russia, Ecole des Mines d'Albi-Carmaux, France and University of Veszprem, Research Institute of Chemical and Process Engineering, Hungary.

<u>The objective of the study</u> is improvement of prediction authenticity of theoretical methods by application of "up-to date" mathematical methods to simulate mixing process in static mixers of different operation principles. The aim is an application of the data obtained to development of methods for static mixer calculation (design). In order to work on our objective, the following plan will be followed.

Firstly, we will analyze the current situation in mathematical simulation, experimental investigation and technological calculation of batch and continuous mixing characteristics in chapter I. We will try to develop a scheme of mixer classification, according to the initial state of materials and the means of agitating force. It would be also interesting to consider some advantages and disadvantages of different mixer types including static mixers.

We will regard the laboratory equipment, materials used for experimentation in chapter II. The methods of experimentation will be also developed for obtaining more accurate results. These data will be used for identifying the model parameters and comparing with the calculated results.

Having the experimental results, we will develop the one and two dimensional models based on the theory of Markov chains in chapter III. To solve this problem, algorithms of building transition probability matrix for one and two dimensional chain will be described and an approach to transformation of microstates to macrostates will be proposed. The problem of initial position of components in the upper loading

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container will be investigated. Then, we will draw some graphs how mixing conditions influence mixture quality.

In order to check if the models work properly, we will compare the results of empirical experiments and simulation in chapter IV. Finally, the algorithms of model parameter identification of both one and two dimensional chain models, the list of experiments required for model parameter identification, the conditions limiting throughput of such type mixers will be formulated and the problem of optimal loading the upper container will be proposed.

CHAPTER I:

STATE OF THE ART IN MIXING TECHNOLOGY AND ITS MATHEMATICAL SIMULATION

1 SAMPLING SIZE. CRITERIA OF THE MIXTURE STATE. MIXTURE QUALITY

1.1 Sampling size

One of the most representative problems in mixing analysis is a sampling size. In some applications, the number of samples taken N can be quite usefully linked to a certain size of the product, say the size of a bag or the size of a pill. Then this size is used for taking samples. However, it overlooks the internal structure within the scale of scrutiny [9].

A particularly useful concept developed by Hersey [10] is the "ordered mixture". According to the concept, the coarser component of mixture is coated by a finer one with the bonds between particles of different type, holding the particles in a constant relative position. The key to success in this approach lies on the correct control of the surface chemistry of the components. As far as there is difficulty in making accurate control of the surface, lots of on-line sampling of powders is not practiced.

Theoretical means of describing the microscopic structure of mixture have been available for some years coming from the early work of Danckwerts [46] but the problems of sampling and data analysis have led to it being little used. Advances in image analysis and rapid data processing make it more feasible now.

In spite of these problems, it is necessary to define a degree of mixing to be able to estimate mixing process. Concentration of components in a sample is often used as a mixture characteristic. Thus, the sampling size represents the resolution by which mixture can be judged. The smaller the size of the sample is, the more



closely the condition of the mixture will be scrutinized (see figure I-1). Dankwerts [46] calls it the "scale of scrutiny". However, it is not possible to diminish the sample size unlimitedly as it is restricted at least by particle sizes that form the mixture. If the sample size is the whole volume, any mixture is certainly homogeneous and if the sample size is a particle,

Figure I-1: Sampling size.

any mixture is evidently non-homogeneous.

Therefore, specifying sample size is an essential step in analyzing mixture quality. This should be specified in connection with further mixture application. In pharmaceutical industry there must be a guarantee that active ingredients are evenly distributed, e.g. within the individual tablets. This is less critical for mixing materials in some industry, where the sample size can even be in tons.

1.2 Criteria of mixture quality

Mixture quality is determined by the performance of the whole group of items of equipment that make up the plant [9]. It generally includes the storage hoppers, conveying system, the mixer itself, the mixer discharge system, and the final piece of plant that produces the final product: a packet, a compact or whatever final form is required. There are many different approaches [1, 2, 3, 43, 48, 69] to determine mixture quality, what makes it difficult to analyze and compare the obtained results.

In most studies reported in the literature [29, 30, 36, 37] the sample concentrations have been taken to calculate their variance, hopefully of stated size, and some function of the variance has been used to describe mixture quality. This way of describing the structure of a mixture conceals certain information and does not take into considerations other items. Thus the information about the internal structure of the samples is lost and the information on the actual co-ordinate positions from which the samples are taken is simply ignored. There are considerable challenges in obtaining sufficient information in order to find variance accurately. About 40 samples have been recommended [3], but it usually imposes significant problems in analysis and, thus less number of samples is usually taken.

As far as mixing is a random process, estimation of the mixture quality obtained is based upon the methods of statistical interpretation. In order to estimate mixture quality by one random value, the mixture is considered to be bi-component (see figure I-2). One component is assumed as being a key one and the other components are combined into a second component. Therefore, having distribution of the key component in the second conventional component, it is possible to examine mixture quality. Hence, concentration of the key component in micro volumes is a random value X for the bi-component mixture. This random discrete value is entirely characterized by distribution laws, mathematical expectation, variance or standard deviation, and sometimes moments of high order [2].



Figure I-2: Illustration of the homogeneity change in a bi-component mixture (a- totally separated, b - transition state, c - random mixture, d - ideal mixture.

The most common way of estimating mixture quality is considered to be the standard deviation of the concentration of the key component in the samples taken from the mixture. Standard deviation S is calculated from the experimental results according to the following equation:

$$S = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (X_i - \langle X \rangle)^2}$$
 (I - 1)

where X_i is the random value X in the i-th experiment, in our case it is the concentration of the key component in the i-th sample; <X> is arithmetic mean of the values X obtained, in our case it is the arithmetic mean of the concentration of the key component in all the samples taken; n is the total number of the samples taken.

If the number of samples is high, the value <X> tends to the mathematical expectation M of the random value X. Standard deviation S depends on the value <X> and S has its size.

There are also different criteria of determining mixture quality as mixing degree, relative dispersion, reduced standard deviation, etc. In this work, variance and concentration distributions are used to judge the mixture. The variance is calculated by

$$\sigma^{2} = \frac{1}{n-1} \sum_{i=1}^{n} (c_{i} - \langle c \rangle)^{2} , \qquad (I-2)$$

where n is the number of samples taken; c_i is the concentration value in the i-th sample, <c> is the average concentration in the volume.

The objective of continuous mixing is to reduce the mass fluctuations of the key component at the mixer inlet. Here, the main characteristic is gear-ratio of variance oscillation at the mixer inlet and variance oscillation at the mixer outlet (Danckwerts [46]). This characteristic is called VRR (Variance Reduction Ratio)

$$VRR = \frac{\sigma_{in}^2}{\sigma_{out}^2} \tag{I-3}$$

Variance reduction ratio is more frequently used than other criteria because it allows qualifying effectiveness of the mixer. Another criterion that is frequently used in industry to estimate the mixture quality is a coefficient of variation (CV). It is calculated according to the following equation:

$$CV = \frac{\sqrt{\sigma^2}}{\mu} \cdot 100\% \tag{I-4}$$

where μ is the average concentration.

2 MECHANISMS OF MIXING

Understanding mixing mechanisms helps to control, Gyenis [32]. Mixing mechanism is closely related to the way of relative displacements of various parts and constituents of the bulk solids. Lacey [9] proposed three different kinds of mechanisms, namely:

1. convective mixing is a transport of large particle groups from one location to another (it is quick but not effective at small scale);

2. *diffusive mixing* is defined as distribution of particles over a freshly developed surface (it is long but effective at small scale);

3. shear mixing is a set up of slipping planes within the mixture.

However, we can suppose that shear mixing is a case of convective mixing.

Diffusive-type mixing may take place not only on free surfaces, but also within the bulk of particle beds. It also should be noticed that behavior of a real mixer is much more complicated and it can not be described by only one of these simple mechanisms. It can be stated that several mechanisms act simultaneously in the majority of bulk solids. Thus, convective and shear mechanisms can not be totally separated from each other, and their interrelation depends on the actual conditions and on the properties of treated materials. In loose particle bed, e.g. in pneumatic conveying, convection is generally accompanied by relatively low velocity gradient and mild shear only. Having turbulent flows, such as in mechanically fluidized particle beds, velocity gradient can be considerably higher, but shear occurs mainly between



Figure I-3: Diffusive and convective mixing mechanisms.

neighboring particles [25]. In dense particle beds, convection and shear occur together, especially if considerable velocity difference exists between the adjacent zones. Dense regions and velocity differences are always present in the majority of mixers and thus convection and shear mechanisms act simultaneously (figure I-3).

Diffusive mixing is analogous to molecular diffusion, taking place in fluids, with respect to the random walk of particles. The principal difference is that molecular diffusion takes place spontaneously, while particles should be agitated to move.

Diffusive and convective-shear mechanisms usually act together within a mixer. For example, convection and diffusion can occur simultaneously in mechanically fluidized beds along the whole reactor volume. In rotated drum mixers, diffusion-type mixing takes place mainly on the upper free surface where the particles are rolling down on the slope, accordingly to the dynamic angle of repose. At the same time, convection takes place in the particle bed. Some individual particles may change their regular paths between these streamlines, causing certain diffusive-type random walk inside the particle bed. These mechanisms, acting together, result in strong transversal and relatively weak axial mixing.

Mixing processes can also be distinguished according to their systematic or stochastic feature [9]. In a macroscopic sense, stochastic mixing is a process where different regions of a particle bed are rearranged at random. It corresponds to a chaotic convection-shear mechanism. In the microscopic sense, stochastic mixing is identical to the diffusive mixing caused by chaotic movements of individual particles. Shear and convection mechanisms are peculiar to certain types of blending silos and motionless mixers, where the streams of material are repeatedly divided and rearranged in space and time, according to predetermined regular patterns.



To perform a really efficient mixing, simultaneous action of different mechanisms is desirable. Convection and shear alone cannot produce a high degree of homogeneity at the particle level within a reasonable time. At early mixing stages (if mixture is completely non-homogeneous) convection mechanism runs more efficiently than the diffusive or dispersion

mechanism. Later on diffusive mixing becomes more efficient than convection-shear mechanism (see figure I-4). Therefore, if diffusive mixing acts together with convection and shear, it has a strong synergetic effect, making it possible to achieve high mixture quality within a short time, Bridgwater [9].

<u>3 MECHANISMS OF SEGREGATION</u>

Segregation can be defined as the inverse process of homogenization [32]. Almost uniform spatial distribution of components might transform towards the segregated state in a non-stirred but not "frozen" particle beds, or in a mixer where segregation has more influence than mixing. It means that segregation may occur even during mixing operation, competing with the process of homogenization. Segregation of particulate materials can result from different reasons. Williams [11] and Weinekotter [3] described several different reasons of segregation:

1. *Trajectory segregation.* It is caused by the deviations between the trajectories of particles traveling through a stationary or streaming gas phase, or by different paths of individual particles, rolling or sliding down on the inclined surface of the particle bed (see figure I-5c).

2. *Percolation segregation* means that fine particles migrate through the holes between the coarser ones. Percolation is enhanced by shaking, vibration, or by the flow of the particle bed, opening new and new gaps temporarily (see figure I-5b).

3. *Floating in particle beds* [3] leads to quite similar results as percolation, but in this case the coarser particles are floating up to the upper surface of the particle bed. It results from the fact that smaller particles are penetrating into the holes just below the coarser ones, then pushing them upwards due to wedging. New holes open due to shaking, vibration or another kind of motion and this mechanism runs again. Percolation takes place mainly in particle beds where the volume fraction of fine particles is relatively low, while floating mechanism acts when the coarse particles are in minority (see figure I-5a).

Floating or sinking [3] may act in fluidized bed or during two phase gas-solid flow. The relative sinking or rising behavior depends on particle size, density, shape,



Figure I-5: Illustration of some segregation mechanisms.

surface properties, the relative velocities of the solid and gas phases, the void fraction of the particle bed, and other factors.

Besides these basic mechanisms, certain special forms of segregation were also described by several workers. Shinohara and Miyata [12] proposed the term of *screening mechanism* acting in an inclined flow of multi-component particle bed containing species of different sizes. This mechanism is similar to percolation, because fines are screened through a mixture layer, and thus they are settling towards the bottom. The so called *flow pattern segregation* described by Shinohara and Enstad [12] is the consequence of a non uniform cross-sectional flow, e.g. funnel flow, during discharge from a container or silo. Particles starting from the central region will leave the vessel first, later on followed by particles coming from the periphery regions of

the vessel. Comparing this composed mechanism to percolation mentioned above, principally little difference can be found between them.

In the majority of bulk solids handling, several segregation mechanisms may act simultaneously or separately, i.e. at different times or in various regions of the particle bed. This statement is also valid for bulk solid mixing, where segregation increases the required operation time and decreases the achievable degree of homogeneity. Trajectory segregation can take place on the free surface of dense particle beds, as well as flow pattern segregation, screening and percolation mechanisms. Inter-particle percolation, screening and trajectory segregation are the most peculiar mechanisms to rotating drum mixers [32].

Floating mechanism and trajectory segregation may have significant role in loose particle beds [3]. Therefore, these effects have to be taken into consideration and they should be possibly counterbalanced in fluidized beds and pneumatic mixers. The video prints in figure I-6 show an extreme example of segregation [3]. A plexiglas drum is filled with two fractions of monosized Polyethylene bills, which differ by color and slightly in size. Before mixing, the mixture is already quite well premixed (see figure I-6 top). However, while mixing, areas of one or several ingredients form stripes running perpendicular to the mixing axis (see figure I-6 bottom).



Figure I-6: Banded segregation in a drum caused by differences in particle size top: The components are quite well premixed before the mixer is started; **bottom left:** the mixers rotates slowly, no cascading effect; after some time banded segregation occurs; **bottom right:** segregation completed. Videoprints derived from: BASF- Product Properties and Process Engineering [3].

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Finally, the mixture is nearly completely segregated. The drum rotates slowly with very limited convective action so that particles roll on each other and "feel" their difference in size.

There are still lots of challenges in studying segregation. Williams gives an overview of the literature on the subject [11] and suggests the following measures to counter segregation:

- Adding a little water forms water bridges between the particles, reducing their mobility and thus stabilizing the condition of the mixture. Because of the cohesive behaviour of particles smaller than 30 microns, the tendency to segregate decreases below this grain size.

- Inclined planes, where particles can roll, should be avoided.

- In general, having ingredients of a uniform grain size is an advantage in blending.

- Multi-component mixture reduces risks of segregation by "blocking the holes".

<u>4 CLASSIFICATION OF MIXING PROCESS AND TYPES OF MIXERS ACCORD-</u> <u>ING TO DIFFERENT CRITERIA</u>

4.1 General characteristics of particulate solid mixing

The objective of any mixing process is to obtain perfectly homogeneous mixture of two or more components [1]. In order to perform mixing we have to fulfill two requirements. At first, it is necessary to create the boundary between the components, in other words we have to provide a contact between the particles to be mixed. Secondly, it is necessary to imply agitation force that will induces motion of particles. Different mixers fulfill these two requirements differently. Let us regard some approaches providing the requirements.

1. The boundary between powders can be simply organized by placing them in a volume.

- Here batch mixing is characterized by the step loading of material. After having been loaded, mixer starts working and the ready made mixture is unloaded after mixing is finished. This mixer should produce homogeneous mixture in a volume in all directions at the desired scale of scrutiny (figure I-7a).
- Continuous mixing is characterized by constant loading and discharging. While classifying continuous mixers, the fundamental importance is an orienta-





Figure I-7: A sketch to classification of the mixing processes and mixers.

tion of the particle mobility. If particle mobility takes place only in crosswise direction, then effective mixing occurs in cross section of the apparatus (figure I-7b). Particle mobility can exist both in crosswise and axial directions and mixing will take place in both directions (figure I-7c).

2. To create particle motion, agitation force is implied to overcome the threshold friction. It can be attained by rotating the volume, moving blades inside the volume, inducing vibration, organizing free fall of particles, etc (figure I-7 at the bottom). Mobility of particles depends on their physical properties as density, size, shape, etc. Thus, there are various types of mixer designs which use different ways of particle agitation.

4.2 Classification of mixing and mixer types

Classification of mixer can be done according to different criteria, for example according to the design (horizontal mixers, vertical mixers and mixers with shaft rotation, one sectional mixers, two-sectional mixers, etc.). This method of classification almost does not bring anything about the essence of mixing process, that it is rarely used. Another method of classification is based upon the common features of the process (mixers with fluidized beds mixers with loose particle beds (extruders), etc). This method is convenient sometimes from the point of view of common mixing mechanisms, but it does not propose a wide outlook on the great variety of mixers.

Classification according to the state of components in the operating zone and agitating force is considered to be more appropriate and visual. After developing the sketch proposed in figure I-7, another type of classification can be suggested (see figure I-8. According to this scheme, mixing can be batch and continuous. Each of these types can apply different agitation force. Therefore, particles can move naturally by gravity force or they can be agitated to move by vibrating, pushing, stirring, etc. Thus, any mixer can be related to some class according to these two principles. For example, alternately revolving static mixer SysMix can be related to mixing in a volume (batch type) where particles are induced by gravity force. Continuous blade mixer can be referred to Flows (continuous mixing) where agitation power is used.

Let us briefly regard some mixers of different design, with a special emphasis on static mixers that will be under research in this thesis.



Figure I-8: The scheme of classification.

4.3 Mixers with agitating force

Mixer Gericke MXC-150 can be referred to the batch class where mixing occurs in a volume. Particles are agitated by blades, rotating inside the volume. Firstly, the components are loaded into the mixer and mixing starts. Then the mixer is stopped and the ready made mixture is discharged (figure I-9a).

Drum mixers can be referred to the class of continuous mixers (see figure I-9b) where particles are agitated to move. The drum mixers are usually produced in a cylindrical frame being horizontal or inclined with the angle up to 4°. The drum is rotated by a motor and the mixture is constantly discharged from the other part of the mixer.

The Vibro-mixer shown in figure I-9c is related to continuous mixers. The tube is moved highly accelerated along circle or ellipse. These accelerations are so high that the mass of material comes off the walls at one moment and it contacts with the walls at another moment. The large part of material circulates in the cross-sections of the tube in the opposite direction of the vibrator rotation. Axial motion of the mixture and mixing itself results from the difference in heights of layers at the place of loading and discharging.

The screw blade mixer can be also referred to continuous mixers (see figure I-9d). The rotation of the shafts is not higher than 150 rpm. The shafts are rotated by electrical motor connected to the reduction gear. Double shaft mixers have one drive shaft directly bundled with the reduction gear. The other shaft is rotated by a pair of gear wheels.

The vibro-mixer DVS-N shown in figure I-9c,e can be related to the class of continuous mixers. The mixer has two shafts with blades and they are rotated in opposite directions. Some blades have inclination of 90° and others have 45°. The first group of blades has a function of mixing and the last group has a function of pushing material along the mixer body.

4.4 Mixers with gravity force. Static mixers

Mixers where particles are brought in motion by gravity are also widely applied in food processing industry to add species, mineral matters, emulsifying agents, etc.



Figure I-9: Mixers where particles are forced to move by blades, vibration etc; a) batch mixer Gericke MXC-150, b) continuous drum mixer; c) pipe vibro-mixer; d) screw-blade mixer; e) vibro-mixer DVS-N.

Other area of their application can be found in production of cereals, yoghurts, and in homogenization of suspension mass flows.

Continuous static mixers can be related to the continuous class of mixers where the agitating force is gravity. As an example, the gravitational chute mixer can be referred to this group of mixers (figure I-10a). The components are mixed due to motion of material induced by gravity force in the gravitational chute mixer. The following designs of such mixers are well known: chute, silo, shock-and-pulverizing, vibro-gravitational. The material flow rearranges as soon as it faces the chute surface. The material slides along the chutes with a thin layer. Components are mixed when they come to one point from the first two chutes and when they irregularly move from one chute to another.

The continuous gravity conical mixer relates to the continuous class of mixers. It is more effective than the one mentioned above due to better particle rearrangements. The scheme of the mixer is shown in figure I-10b. The components are mixed due to irregular particle motion like in "smaller silos".

Alternately revolving static mixer shown in figure I-10c can be referred to the the class of batch mixers. The operational principle of this mixer type is as follows [25-32]. The components to be mixed are loaded into the lower container i, and afterwards the mixer is rotated along the axis (iii). Hence, the material goes through the mixing zone due to free fall (iv). Afterwards the mixture again comes to the container (i) and the mixer is rotated the necessary number of times to reach the mixture quality required. Mixing occurs both in axial and crosswise directions. Finally, mixture is discharged from the lower container (i).

Continuous static mixers shown in figure I-10d are related to the class of continuous mixers [33-35]. The mixer consists of the following parts: the mixing zone (a tube where static mixer elements are installed) and a mixture collector that can be an apparatus in the next processing stage [34]. Mixing mainly occurs in crosswise direction in such a mixer type. Static mixer elements can be made from winding stripes (see figure I-10e). The interconnection of the stripes can be made with the angle of 90°.







SM







Figure I-10: Mixers with gravity force used; a) Continuous gravity gutter mixer; b) continuous gravity conical mixer; c) alternately revolving batch static mixer SysMix; d)various static mixer produced by Sulzer company, e) static mixing elements of continuous mixers.

5 APPROACHES TO SIMULATION OF MIXING. BASIC CLASSIFICATION

5.1 The scale of modeling. Lagrangian and Eulerian approach

When dealing with particle flow modeling or with powder mixing, one has now two strategies to represent the motion of the particles (Gyenis and Szepolgyi [59]): Lagrangian models or Eulerian models.

1. The Lagrangian models are based on the analysis of the motion of individual particles under all forces, including particle-particle collisions or particle-wall collisions. This approach experienced a new birth in the past decade, since the generalization of codes developed with the Distinct Element (DEM) or Discrete Particle Simulation (DPS). Some of such results can be currently found in the scientific literature (for example Kaneko and Shiojima [74], or Mc Carthy and Ottino [75]). Their main advantage can be found in the relation with the physics that gives better understanding of the process. It has found applications in gas-solid flow and solid-solid flow. However, there are some limitations for their application in real process and equipment design. A formal one is that the use of DEM models becomes tedious when the number of particles is high (superior to 15 000), which is the case in many processes. There are also some more fundamental limitations. For instance, a variation of initial conditions even for one particle completely changes the picture of the ensemble motion after a very short interval of time. Of course, many of average characteristics retain, but it might be easier to deal with the average characteristics from the very beginning like in thermodynamics. Moreover, in contrast to thermodynamics, all particles of real granular material are different, and it is simply impossible to assign to every particle its individual properties, that must be also measured. It is also problematic to describe precisely the interaction of irregular particles, particularly if such complex phenomena like agglomeration are to be taken into account. Thus, the approach is very physical but still very problematic for practical application.

2. The Eulerian approach deals with the medium of particles and it describes variation of the medium properties while crossing a fixed volume of the medium in a flow. It can be noted that the model of particle medium is also used in the Lagrangian approach while describing motion of gas or liquid, but there we observe motion of the element with a flow. If the volume is infinitely small, the problem can

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be described in terms of differential equations. However, the definition of the infinitely small volume for a medium of real particles is controversial because even a very small particle is much larger than the infinitely small volume. The transition to a finite volume gives us... a Markov chain of an appropriate dimension. The transition to the finite volume is always based on averaging the medium parameters within the volume and property flows through its borders. Sometimes the Eulerian approach applied to this purpose is called a stochastic approach because it deals with stochastic components of particulate phase motion. It is based on the global representation of microscopic phenomena under a mesomacro description, such as a dispersion coefficient, a drift coefficient, or transition probabilities (see for example Orban and Mihalyko [76]). The main advantage is that those models use the scale of the process equipment and computation time is an irrelevant factor. Lack of input of physical entity to calculate transitions can be referred to disadvantages.

As mentioned above, the choice between the two alternatives will basically depend on the objective of modeling, or on what type of criteria will be considered as a satisfactory answer to the problem. In this way, we will use the meso-macro description (scale) of modeling, or in other words, the Eulerian approach in terms of Markov chain theory.

5.2 Models of local and integral characteristics

Different authors [3, 40, 37, 49, 45, 72, 64, 36, 46, etc.] use different characteristics to explain the results of modeling. The scheme of classification of models according to the simulation characteristics is shown in figure I-13. Very often integral



Figure I-13: Classification of models according to the simulation characteristics.

characteristics are employed to describe the mixing process (e.g. kinetic models [37]). It can be variance, degree of segregation, degree of mixing etc. This type of models can well describe this characteristic but the model can say nothing about other local or integral characteristics of the process, for example concentration distribution of the components in a volume. This brings a significant disadvantage to these models because several characteristics of the process are often interesting and it is not possible to get this information from the model.

The other models describe local characteristics of the process. They are more informative and allow calculating all interesting integral parameters required by using local characteristics. For instance, if it is necessary to find variance distribution it is just enough to put the formula into the model and calculate it from the concentration distribution known without changing the model.

Mixing of several components is usually carried out. There are components that do not have tendency to segregation. In this case the steady state will contain homogeneous mixture and the question is how much time is required to reach the homogeneous mixture. The models, working with such components, calculate the residence time distribution (RTD), mean time, and this information will be enough.

If the components have tendency to segregation, the situation becomes much more difficult. While mixing, homogeneity can turn from increase to decrease. It means that the model has to answer the questions what maximal homogeneity is reached and when to stop mixing. It is necessary to note that the value of homogeneity will depend on the initial distribution of the components to be mixed.

5.3. Current situation in mathematical modeling of particulate solid mixing

A real mixer transforms some values of the mixture characteristics to other values of these characteristics. Which characteristics to use depends on certain technological process and requirements applied to determine mixture quality. It might be an integral characteristic (for example, variance of the key component in a mixture) or distribution of the key component in a volume. It should be noted that concentration is necessary to measure in the representative samples of the whole volume. The back-calculation of concentration from variance is not possible because this procedure does not give a single solution. The mathematical model of the real mixer should also transform input characteristics to output ones. Any model is developed to describe certain aspect of a process. That is why models, describing different aspects of the same process, may look very differently. It is the main reason why so many models are developed to simulate mixing process. The basic problems of mixing simulation are discussed in [52, 63, 103], and different approaches to simulation can be found in [3, 40, 37, 49, 45, 72, 64, 36, 46].

As far as we are more interested in the description of mixture state evolution, the spectrum of possible approaches is apparently decreasing. Let us start their analysis from simulation of batch mixing.

It is simpler but more laborious to create models binding integral mixture characteristics at the beginning and at the end of the process. Commonly, their relation can be written, as

$$\boldsymbol{\sigma}_{2}^{2} = \mathbf{f}(\mathbf{p}_{1}, \boldsymbol{\sigma}_{1}^{2}) \tag{I-5}$$

where p_j – is the vector (totality) of the control parameters (e.g., rotation speed of the blades, the shape and the number of blades, filling degree of the useful volume, amplitude and frequency, etc.). Indices 1 and 2 belong to the beginning and the end of the process.

Function f is built in the best way to fit the array of experimental data. If the choice of the function is successful, the adequacy of description can be very high, of course if the experimental data are reliable and reproducible. The model can work only within the variation intervals of control parameters that took place during the experiments. It does not consider the internal structure of the process and it regards the process as a black box or RIRO box (Rubbish in – Rubbish out). "Opening" the RIRO box moves aside this approach on the lower levels of the simulation hierarchy. However, to exclude the RIRO box, while modeling, is not possible. This principle of mixing simulation (I-5) was used in [36, 46].

One of the approaches to "opening" the RIRO box models (I-5) is introduction of time, i.e. transition from initial and final states bound to kinetics of the integral characteristic.

$$\boldsymbol{\sigma}_{2}^{2}(t) = f(\boldsymbol{p}_{1},\boldsymbol{\sigma}_{1}^{2},t)$$

(I-6)

If (I-5) regards time as an additional control parameter, then relation (I-6) remains the same as (I-5), though the possibilities of the model application significantly extend. The situation changes if some hypotheses are made and some equations are written about the evolution of σ^2 , where p_j determines the parameters of these equations.

Such approach was used by L.T. Fan and H.H. Gelves-Arocha [37] proposing a kinetic model of "mixing – segregation" in static mixers. The authors separated the process into two stages, the first one is transformation of segregated components into a mixture state, and the second one is the transformation of mixture state into another segregated state. Light and heavy particles were used to form bicomponent mixture.

In this model, evolution of segregation degree f is calculated for both components according to two differential equations of the first order. One equation is for mixing process and the other one is for segregation process. A final segregation degree is assumed to be a sum of individual solutions of these two differential equations. It is possible to vary the moment of time when the mechanism of segregation starts taking place and with the two fitting coefficients K'₁ and K'₂. It is not possible to determine concentration distribution in the collector of material because an integral characteristic (degree of segregation) is considered in this model. This can be regarded as a disadvantage of the model. One more disadvantage is that the model does not have any links with the real equipment. For instance, if the number of mixing elements changes, the fitting coefficients have to be identified once again what requires additional experimental work. The model is coordinate continuous, what brings difference in determination of physical and infinite small volume.

Notwithstanding the undoubted practical value of the models operated with the integral characteristics, more informative models are those describing evolution of extended characteristics, for instance, evolution of the key component concentration [3, 40, 41, 69, 70, 71]. Though these models have superficial difference, in the end they use the same dispersion equation (or its differential representations), that is

$$\frac{\partial c}{\partial t} = \operatorname{div}(-c \cdot \upsilon + D \cdot \operatorname{gradc}) \tag{I-7}$$

According to the interpretation of the variables, different names are given for this equation. If c(r,t) has a sense of probability density distribution of the key component, then v is called the transport coefficient, D is the stochastic parameter, and the equation has the name of Kolmogorof's or Fokker-Planck [101], (probabilistic or stochastic model). If c has the sense of concentration, then v is called the velocity of convection, D is the coefficient of dispersion, and the approach itself is called the dispersion model [3]. Besides, work [20] gives strict evidence that these two interpretations are equivalent.

Although the theory has formulas for calculating υ and D, they are more likely to explain their nature, than to be used for calculation. It results from their operation with the variables being more difficult to identify from the empirical experiments than the original υ and D.

Equation (I-7) relates to the so called continuous approach, where differentiation operations in coordinates are determined. Applying the approach to powder handling and processing, it should be recognized as a "model" one, because all the local perturbations (e.g., blade motion) are "spread" along the visible working volume and are hidden in the values of u and D.

One dimensional dispersion equation is often used. It is written for the determining coordinate of the process, e.g., axis x

$$\frac{\partial c}{\partial t} = -\frac{\partial}{\partial x} (c \cdot v) + \frac{\partial}{\partial x} D \frac{\partial c}{\partial x} , \qquad (I-8)$$

and more often its linear case when it is assumed that υ , D=const

$$\frac{\partial c}{\partial t} = -\upsilon \frac{\partial c}{\partial x} + D \frac{\partial^2 c}{\partial x^2}$$
(I-9)

It relates to the parabolic partial differential equations, the solution of which has been very well investigated [67].

If the boundary conditions at the edges of the fragment [0, I] are accepted as

$$\frac{\partial}{\partial x} \left(D \frac{\partial c}{\partial x} - c \cdot v \right) \Big|_{x=0,1} = 0, \qquad (I-10)$$

(what means that the particle flow is not present), then the equation simulates batch mixing with conservation of mass of the key component within the fragment. This approach was used in [19]. It is necessary to note that asymptotically homogeneous distribution is achieved only if v = 0, i.e., particles do not have a preferred motion di-

rection in the volume (particles do not have tendency to segregation). If $\upsilon \neq 0$ (segregation exists), the mixture evolution character depends on the initial distribution and mixture quality can have a maximum in time. It means that the process should be stopped at that point [18]. It is worth saying that the assumption on the process linearity is appropriate if mass of the key component is relatively low (in fact, when it plays the role of a tracer). If its mass is high, particles of the key component do not penetrate into the base component but its mixture with the key one. It can significantly influence the values of υ and D. If that is the case, equation (I-8) should be applied with $\upsilon=\upsilon(c)$ and D=D(c), which does not leave any chance to find analytical solutions.

To simulate one dimensional continuous mixing, the edges of the fragment [0, I] (or one of them) are open. Consequently, the function of external source q_e is introduced in (I-9)

$$\frac{\partial c}{\partial t} = -\upsilon \frac{\partial c}{\partial x} + D \frac{\partial^2 c}{\partial x^2} + q_e(x, t)$$
(I-11)

 $q_e(x,t)$ is usually localized in the cross-section of material feed (e.g., near the edge x=0) in the point or on the fragment, and the boundary condition of the third kind is set up for the right boundary x=l

$$(\mathbf{c} \cdot \boldsymbol{\upsilon} - \mathbf{D} \frac{\partial \mathbf{c}}{\partial \mathbf{x}})\Big|_{\mathbf{x}=\mathbf{l}} = \boldsymbol{\upsilon}_{\mathbf{l}} \mathbf{c}\Big|_{\mathbf{l}},$$
 (I-12)

where u_l is the outflow velocity of material through the fragment. It reflects the characteristic of the discharge device.

If equation (I-11) is used for the description of continuous mixing, material is assumed to be mixed homogeneously in the direction perpendicular to the axis x. Then the subject of analysis is the relation between inflow and outflow fluctuations. This relation can be obtained both from the solution of equation (I-11) (the examples of these solutions are given in [69]), and from the residence time distribution of particles after an impulse tracer injection.

$$q_e(x,t) = q_0(x) \cdot \delta(t) \tag{I-13}$$

The RTD is the function of response after the impulse injection, and in the case of this model, it will be

$$\mathbf{E}(t) = (\mathbf{v} \cdot \mathbf{c}(t) - \mathbf{D} \frac{\partial \mathbf{c}(t)}{\partial \mathbf{x}})\big|_{\mathbf{x}=\mathbf{1}}$$

 $\left. c(t) - D \frac{\partial v(t)}{\partial x} \right|_{x=1} \tag{I-14}$

The RTD has been obtained by different authors [66] from analytical solution of equation (I-11). RTD is numerically characterized by its moments, such as the first order moment which is the mean residence time of particles.

Continuous approach on the basis of equation (I-11) was used in [40, 42, 69, 70, 71] to simulate mixing. Authors used two equations (I-11), each of them was written for its own component. The motion of components was modeled independently from each other. Afterwards, a separate equation of mass balance was used. However, it does not bring any novelty to the model because just different values of u and D will return the same results that can be obtained from the classical model.

Authors, using continuous approach, usually limit simulation by the cases when it is possible to derive analytical solutions. As far as all possible variants are already obtained, they concentrate on the relation between u and D and the conditions of the process applying sometimes RIRO box models or employing physical phenomena.

6 MODELING BY MEANS OF MARKOV CHAIN THEORY

6.1 General information about the Markov chains

If a more detailed research on the process is required than the analytical solutions allow, it is necessary to involve numerical methods. In our point of view, the most visible and convenient approach for writing balance equations is based on cell models and models employing the theory of Markov chains [36, 46, 47, 52, 61, 64-68, 94-107].

Although the theory of Markov chains was used for simulating mixing process by many authors [36, 38, 46, 47, 52, 61, 64-68, 94-107], more or less universal strategy to application of the theory to processes with particulate solids was described in [64, 18]. The basic idea of one dimensional model building is shown in figure I-14. The interval of the property is divided into a finite number of discrete intervals n, which are called system states. In this case, probability density distribution function is replaced by the probabilities of the state S_i . All possible states are transformed into the state space. The set of their probabilities is the state vector S with the size m×1:
$$\mathbf{S} = \begin{bmatrix} \mathbf{S}_1 \\ \mathbf{S}_2 \\ \dots \\ \mathbf{S}_m \end{bmatrix} = \begin{bmatrix} \mathbf{S}_1 & \mathbf{S}_2 & \dots & \mathbf{S}_m \end{bmatrix}$$
(I-15)

where ' means transposing a vector or a matrix.

 S_i can be interpreted for high number of particles as a relative part of particles belonging to the state i.

The total duration of the process can also be divided into finite time intervals Δt , and the current time can be expressed as a sequence of discrete values $t_z=\Delta t(z-1), z=1,2,...$ It allows us to substitute continuous time by the integer numbers z. Thus, by using this approach all the variables become integer ones.

The state vector S_z changes and becomes S_{z+1} during z-th transition. It means that particle property also changes and it can transit to another interval of this property. An example of possible transitions is marked with arrows (see figure I-14). Every transition has its own probability. Probability also means the part of particles in a cell, that transits to the corresponding direction. The value P_{ij} means the part of particles in the cell j, that transits to the cell i. If i=j, then P_{ij} is the probability of the property to remain in the cell during the time interval of one transition.

The relation between S_z and S_{z+1} can be expressed by the matrix equation:

$$S_{z+1} = PS_z \tag{I-16}$$

where P is the matrix of transition probabilities, or the transition matrix (the basic operator of the model on the basis of the Markov chain). It consists of transition probabilities and it can be constructed according to the following principle: j-th column of the matrix consists of the probabilities belonging to the j-th cell. The probability to transit to the i-th cell are placed in the i-th row of this column (I-17).

$$\mathbf{P} = \begin{bmatrix} p_{11} & p_{12} & 0 & 0 & \dots & 0 & 0 \\ p_{21} & p_{22} & p_{23} & 0 & \dots & 0 & 0 \\ 0 & p_{32} & p_{33} & p_{34} & \dots & 0 & 0 \\ 0 & 0 & p_{43} & p_{44} & \dots & 0 & 0 \\ \dots & \dots & \dots & \dots & \dots & \dots & 0 \\ 0 & 0 & 0 & 0 & \dots & p_{m-1,m-1} & p_{m-1,m} \\ 0 & 0 & 0 & 0 & \dots & p_{m,m-1} & p_{mm} \end{bmatrix}$$
(I-17)

In general case, there are two restrictions on the matrix P_{ij} , that are followed from the mathematical principle of the problem:

$$0 \le \mathbf{P}_{ij} \le 1 \tag{I-18}$$

$$\sum_{i=1}^{n} P_{ij} = 1, \quad j=1,2,...,m$$
 (I-19)

The subsequent use of (I-16) allows discribing process evolution in time, if the initial state vector S_0 is known. If the matrix does not depend on the state and time, the process is linear and its evolution can be defined as:

$$S_z = P^z S_0 \tag{I-20}$$

Fot the asymptotical state vector S_{∞} , if it exists, (I-16) yields $S_{\infty}=PS_{\infty}$, and it means that all the elements of the vector S_{∞} are proportional to the vector elements before it is changed by the matrix P. These vectors are called eigen vectors of the matrix P and the proportional coefficients are eigen values of the matrix. Therefore, S_{∞} is the eigen vector of the matrix P, that corresponds to the eigen value equal to 1. It is necessary to say that the asymptotical vector does not depend on the initial state vector.

If particles are introduced into the system after every transition and the initial distribution is known, the process is descrided as follows:

$$S_{z+1} = P(S_z + S_f)$$
 (I-21)

where S_f is the feed vector.



Figure I-14: Transition from continuous distribution to the Markov chain.

Computational procedure (I-21) is always stable. It allows calculating almost all characteristics of the process with particulate solids if the matrix P is known. It can be done by using modern computer software like MATLAB. Therefore, the problem is a consideration of the process physics, development of the cell model and construction of the transition probability matrix.

The choice of the number of cells (or their length with fixed total length) and duration of the transition time are the matter of principle. If we simply like to use the chain model for numerical solution of the parabolic equation, e.g., (I-11), then the more cells are taken and the less the transition time is – the closer we approach the "exact" analytical solution. If to use Markov chains, the number of cells is chosen according to the size of sampling.

Transition time is also a very important problem to discuss. If all the chain states can be reached and transition time is high enough, arrows of possible transitions bind all the cells, and the matrix P becomes entirely filled. If the transition time is the process time (for example, passage of the last portion of components through a static mixer), then the matrix P will bind initial and final mixture states. In other words, the matrix analogue of equation (I-5), where the mixture is not represented by one but

finite number of parameters. This approach was used in [46-48]. Having obvious practical value, this method steps aside the mixture evolution from the initial state to the final one. It automatically excludes the possibility to influence evolution.

Another approach is as follows: the transition time is taken so short that particles can move only to the neighboring cells, not further. In this case the matrix P has three diagonals. Probabilities to remain in a cell are placed on the main diagonal during the time Δt , the lower diagonal is responsible for the probabilities to go forward and the higher diagonal is in charge of probabilities to go in the backward direction. This method allows describing evolution of the process with time. It gives us an opportunity to make technological conclusions.

Application of the Markov chain theory has many advantages. First of all, it can be regarded as a universal strategy for creating models, not only for mixing but also for other processes of chemical technology [17, 18]. The states can represent different physical characteristics of the process. Secondly, the elementary volumes are finite what corresponds to real experiments because physical samples are always limited volumes. Thirdly, the theory is very visual and writing down cells and material motion arrows easily allow writing the matrix of transition probabilities. The theory does not require advanced mathematical skills and it only includes multiplication of matrices and vectors. This matter makes the theory very convenient for its computer processing. As for disadvantages, this theory does not allow looking at what happens inside the cells. It does not describe behaviour of single particles and their interactions. To computational difficulties we can refer keeping the condition of normalization and taking care of the positive values of transition probabilities while describing non-linear phenomena.

This approach has been successfully used for simulation of one and two dimensional continuous mixing, simulation of grinding with different fraction velocities in a mill [17]. It is the approach that forms the theoretical foundation of this thesis.

6.2 Simulation of continuous flows

Oyama and Ayaki [89] were the first who made a research on mixing simulation by Markov chain theory. They studied mixing of different particles with a 2.5 size ratio in a horizontal lab-size tumbler mixer, taking into consideration the effect of initial component loading. The evaluation of the concentration was carried out in each section

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of the mixer volume by sampling, so that in the end sampling errors caused high uncertainties on the transition probability determination. Similar type of mixer was used by Fan and Shin [55] later for the binary mixing of spherical lucite particles with equal densities but different particle sizes (4.8 mm with 3.2 mm or 2.9 mm). Considering that mixing occurs according to Kolmogorov's equation, the authors developed a discrete formalism for the drift and diffusion coefficients, which they linked with transition probabilities of the Markov chain. During the experiments, the mixer was divided into 10 equal sections, and the key component (the smallest particles) was placed in a certain cell for each particulate system. The experiment was repeated two times for each cell without sampling so that a one-step transition matrix could be determined. This matrix was tridiagonal and corresponded to 15 revolutions of the mixer with a slow evolution of the mixing process. Drift and diffusion coefficients were determined from linear regression to time zero and their variation along the axis of the mixer was studied for each particulate system. Having done the experiments, the authors supposed that the diffusion coefficient is constant for the binary mixture, and thus they made a conclusion on the relevance of the axial drift velocity to explain band formation in the mixer. As for simulation, the Markov chain allowed representing the concentration profiles for each system quite well. Some discrepancies are stated to be attributed to the fact that the chain was homogeneous. This work is very valuable but the analysis has been restricted to horizontal tumbler mixers which are neglected in industry since a long time ago as they cause segregation.

The concept of static mixing was developed and examined by Wang and Fan [36, 46] both theoretically and experimentally. Mathematical models developed in these papers were also based on the theory of Markov chains but approaches to representation of the process were different. In [36] the state of particulate system was represented as several microstates (samples) at the top and at the bottom, and the matrix of transition probabilities connected these states after one passage of particles through the mixing zone (see figure I-15). The size of the matrix was equal to the number of the states chosen. The matrix was restored on the basis of experimental distributions of tracer particles at the bottom after putting the tracer in all state positions at the top consequently (see matrix equation I-22). After having been restored the matrix allowed calculating the distribution at the bottom after arbitrary number of passages, of course under the assumption on the linearity of the process. It is obvi-

ous that the model was a black-box model, which was valid only for the mixing zone and the components that were used for the experiments. It is considered to be the main disadvantage because if some condition changes (initial distribution of components, the number of static mixing elements, materials) the model stops giving the appropriate results.

In [46] the authors undertook more detailed decomposition of the process by representing the total transition matrix as a product of matrices related to smaller time steps, when these matrices allowed transitions of a tracer only to the neighboring cells (states) (see matrix equation I-23). However, this approach leads to the same matrix as in the previous paper, and, in fact, does not bring anything new into the process description. The main disadvantage remains the same as in the previous model [36]. There were no suggestions how to take into consideration different properties of components without doing that much of experimental work.



Figure I-15: Schematic representation of the mixing system.

The model proposed by Barantzeva, Marikh, Mizonov and Berthiaux simulates mixing in continuous mixer by the theory of Markov chains [52 63 64 99 103]. A scheme of the mixer and its cell representation is shown in figure I-16. There are blades placed along the mixer length. They interact with particles inside the mixer. The process of blade interaction with particles, particle motion is not clear, so that its theoretical description is still difficult. That is why the motion zone of one blade was taken as a cell. It has been investigated in a stand to obtain relation between blade

parameters and layer parameters (relative part of particles staying in the zone and leaving it in forward and backward directions). To fulfill this task a colored tracer was used [92, 93]. In other words, the process inside the detached zone-cell has been investigated according to the idea of "black box". Then the results obtained were unified to build the mixer model according to the chain approach when the state vector S_k changes to S_{k+1} in a constant period. The continuous time is substituted by its quantified periods $t_k=k\Delta t$, where k is treated as an integer-valued periods of relative time. Correlation between state vectors till the k-th transition and afterwards is calculated by the matrix expression

$$\mathbf{S}_{k+1} = \mathbf{P}(\mathbf{S}_k + \mathbf{S}_f) \tag{I-24}$$

where P is the matrix of transition probabilities.

The model gave satisfactory values of RTD and Variance reduction ratio (VRR) and



Figure I-16: A continuous mixing process and its cell representation.

they were in a good agreement with experiments. This model is both time – discrete and space-discrete. It takes into account motion of material due to action of blades

and does not regard the natural flow due to the inclined surface inside the mixer. This problem was solved in [95, 97,100], where two types of motion were considered: the free flow motion to the decreasing free surface level side and the motion caused by the action of blades.

6.3 Simulation of batch mixing

The pattern approach was firstly used and developed by Inoue and Yamaguchi [108] for a V-type batch tumbler mixer of small width. Supposing perfect symmetry of the flow, half of the equipment (a triangle) was divided into 40 cells drawn in the external surface of the 2D transparent set-up. Glass beads of approximately 2.50 mm diameter were taken as material. A mono-colored bead was added and its position was recorded at every rotation. This allowed analyzing general pattern that mainly consisted of one large circulating flow along the vessel. It resulted in trapping of the particles at the centre and at the edges of the triangle. These data were collected under the matrix form and served to develop a two-dimensional Markov chain model. Some more experiments were also carried out using 100 tracer particles. The obtained results were compared with the simulation results.

Despite this difference that may have led to statistical errors, the agreement between the experimental and calculated results was satisfactory. However, the authors did not discuss some important matters, such as the effect of the flow symmetry, as particles can transit from the left mixer part to the right one.

Much more recently, Aoun [14, 16] proposed a similar experimental methodology for a laboratory hoop mixer. It was divided into 11 axial compartments that were separated into 3 radial cells, thus delimiting 33 elementary cells. They were delimited by placing inserts when positioning the tracer and/or removing it for analysis. Couscous particles of 2 mm diameter were taken as material and a colored tracer was made by steeping couscous particles by iodine. Tracer particles occupied the whole cell volume. Thirty three single step experiments, corresponding to 3 revolutions of the mixer, were performed to build the transition matrix, and the obtained data were used for analyzing flow pattern in the vessel. A Markov chain model (oscillating with reflecting screens) was developed to represent the motion of tracer particles

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in the axial direction, leaving the data concerning radial transitions unused. The flow was represented by two matrices, and each of them corresponded to one half-rotation of the mixer. To make the model simpler, transitions were allowed between the cells separated by no more than one intermediate cell, and probabilities to remain within a cell were all equal during one transition. Two parameters of the model were adjusted to fit the experimental results and the agreement between simulation and experiments was satisfactory. As a perspective, a more sophisticated model can be developed to take into account the data concerning radial transitions.

The stochastic model suggested by F. Delvigne, J. Destain and P.Thonart simulates mixing of fluid and particulate solids in a batch mixer [72]. The model also consists of the initial state vector S_0 , which is multiplied by the transition matrix P (I-23) to give a new state vector S_1 . The relation between the state vectors is defined by the matrix equation

$$\mathbf{S}_{k+1} = \mathbf{P}(\mathbf{S}_k) \tag{I-25}$$

Here, the state vector contained the tracer concentration for all states where a state corresponded to a region of the agitated vessel. The scheme of the process is shown in figure I-17. There are two adjustable parameters for the two-dimensional model: q_e and q_c . The circulation flow rate q_c was calculated by using the equation coming from dimensional analysis:

$$q_{\rm c} = \frac{N_{q_{\rm c}} \cdot N \cdot d^3}{n_{\rm loop}} \tag{I-26}$$

where n_{loop} is the number of circulation loops implemented by the model. N_{qc} is a dimensionless circulation number, which depends only on the impeller geometry in the turbulent flow regime, N is a circulation coefficient.

The turbulence flow rate q_e was estimated by a sensitivity analysis. The value of q_e was modulated for a calculated value of q_c to match the measured mixing time. The maximum and minimum values of q_e were determined to fit the experimental mixing time.

The model described well the concentration variation in time at specific samples in the vessel. The authors showed that Markov chain theory can be applied to studying mixing of liquids and solids. This approach is said to be

CHAPTER I: STATE OF THE ART IN MIXING TECHNOLOGY AND ITS MATHEMATICAL SIMULATION



Figure I-17: Scheme of the model and the matrix of transition probabilities.

used when dealing with complex models involving reactor hydrodynamics and reacting species (such as microorganisms in the presence of nutrient), which require a great amount of computational time and space.

(I-27)

6.4 Modeling flow in fluidized beds

Fox and Fan [23] were first authors who represented a cylindrical fluidized bed as a one dimensional sequence of cells in the axial direction. It corresponds to a Markov chain where the probabilistic matrix equation was employed (Berthiaux [64]). Transitions were allowed from the neighbouring cells and they were all equal, so that the transition matrix was tridiagonal and contained a single parameter. The model parameter was identified using experimental data from the literature. Gupta and Dutta [56] developed a similar axial cell model, using the same experimental data. The transition probabilities were equal between the cells. The authors presented their work on the basis of the theory of Markov chains and calculated the passage times, i.e. transition to adjacent compartment, to find the model parameter. Its value was the same to what was obtained in the previous paper. Though the assumption on equality of upward and backward transitions does not seem to be very real in the upward flow under gravity, these models made a first attempt on modeling fluidized bed by Markov chain theory.

A general Markov chain model has been recently suggested by Dehling [78], Hoffmann and Dehling [54] for simulating residence time distribution (RTD) of particles in a continuous fluidized bed. The upward and backward probabilities are different from one cell to the neighbouring ones and they depend on the position of particles in the bed. The authors introduced an additional probability that allowed taking into consideration the trapping effect in the gas phase that pushes the particles to the top of the bed. This model is more general than the one mentioned above because it considers more flow possibilities for the particles. The authors managed to estimate and find the model parameters as a continuous function of the bed height by using information published on the fluidized bed. The agreement between experimental and theoretical data taken from the literature on the basis of RTD curves is satisfactory. The disadvantage is that the model takes into account only axial mixing.

Harris [87] developed several two-dimensional Markov chain models (a "core annulus" interchange model and a "four-zone" model), considering the radial mixing effect in the bed. The model parameters were bound to local solid flow rates and determined from experiments. It resulted in the absence of adjustable parameter during simulation. The four-zone model gave very compatible results in

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various bed configurations. Therefore, the theory of Markov chains is also a useful tool in solving flow problems in fluidized beds. There are some challenges for future as taking into consideration some more effects, for instance, chemical attrition, mechanical attrition and chemical reaction, what makes the model non-linear.

6.5 Modeling segregation

Segregation can be modeled on the basis of the Markov chain theory (Berthiaux [64]). Wang and Fan [36] proposed a model of segregation in a motionless mixer where they employed a homogeneous Markov chain with transition probabilities fixed during time change. Therefore, this model was linear but as it was pointed out by Chou [84], segregation causes a local change in the mixture structure what affects the transitions and consequently, the model becomes nonlinear.

Afterwards, the authors tried to make an attempt to develop a non-linear Markov chain model to represent segregation in a vertical tube sinusoidally agitated. The tube was divided into 6 removable axial cells that defined the state space of the Markov chain. Binary mixtures of 2.38 and 2.56 mm chrome steel beads were initially mixed to reach homogeneous mixing state, and then the mixture was placed in the experimental device. As far as the largest particles have lower mobility than the smaller ones, the authors suggested that the transitions could only occur from one cell to another one. It is unlikely to happen in reality and the model is desired to be more general. Thus, the related transition matrix contains a low number of unknown parameters that could be determined by solving a linear system of equations (inverse problem).

In further research, tracing experiments were carried out by colouring both components, placing the tracer particles in a given cell and repeating the procedure for all cells. Time was segmented in 4 intervals of 5 seconds each, and the same optimization procedure was implemented. Four related matrices were sensibly different from each other what makes the use of such non-linear chain more valuable. The authors did not advance their analysis comparing the results of a homogeneous chain by examining whether the same state vectors were obtained by multiplying 4 different matrices or by multiplying 4 times the first one (Berthi-

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aux [64]). The evolution of the probabilities with time was not studied, as this could have resulted in a more general model.

Rippie and Chou [83] studied segregation in a sheared particle bed approximately at the same time. A rotary shear cell divided into 36 annular segments of different volume was used. Binary mixtures of steel particles of different size but other equal properties were investigated. The component was initially placed in the mixer in different layers. Similar non-stationary Markov model as above was employed, with a specific correction (under matrix notation) for taking into account the difference in the cell volumes. The tracer experiments were made to restore the model parameters. Both downward and diagonal transitions were set up to 0. Inward transitions were allowed in the radial direction only from the neighboring cells and they were independent of bed depth. Increments of shear during equal times served as a transition of the Markov chain, and the different state vectors obtained were used for calculation. The authors further tried to comment on their results in terms of inter-particle void creation.

7 CONCLUSIONS ON CHAPTER 1

We looked at some mixing mechanisms and criteria of mixing quality used by different authors. Some approaches to simulation of batch and continuous mixing have been regarded with pointing some advantages and disadvantages. Therefore, we will apply the Eulerian approach and try to develop a more general model for static mixers, based on the theory of Markov chains.

CHAPTER II:

LABORATORY EQUIPMENT AND METHODS OF EXPERIMENTATION

Three static mixers of different types were investigated in this thesis. The methods of experimentation with the mixers are similar. In the following we will consider mixer types with various methods of experimentation, materials and supplementary equipment used.

<u>1 THE LABORATORY STATIC MIXER (THE "LAB-MADE" MIXER)</u>

1.1 The mixer concept

The main part of the experimental work was carried out with a specially designed "Lab-made" static mixer that imitates operation of the real alternately revolving static mixer. The mixer (see figure II-1) is constructed from a piece of plexiglas and



Figure II-1: The "Lab-made" mixer.

includes three main zones. The first zone is a feeder separated into 5 sections by removable insertions. The collector of the mixer or the lower unloading container is identical to the feeding part of the mixer. The mixing zone is composed of the holes drilled to put screws into the front panel of the mixer. These screws serve as static mixer elements to make a turbulent flow. The number of screws can be varied from 0 up to 53, and thus the mixing effect depends on the number of screws installed. Polystyrene plugs were used in order to cover the holes, which are not occupied with screws.

The volume of one section of the static mixer is equal to

 $V_{s2} = a \cdot b \cdot h = 20 \cdot 30 \cdot 11 = 6.6 \cdot 10^3 \text{ mm}^3$

It is necessary to make analysis of the space or free volume in the mixer as a function of the number of insertions inside. The whole volume of the empty "Lab-made" mixer is

 $V_{w2} = a \cdot b \cdot h = 20 \cdot 30 \cdot 235 = 141 \cdot 10^3 \text{ mm}^3$



Figure II-2: The "Lab-made" mixer.

The volume of one screw is

 $V_{\text{screw}} = \pi \cdot r^2 \cdot l = \pi \cdot 2^2 \cdot 20 = 251,2 \text{ mm}^3$

Porosity of the the "Lab-made" mixer as a function of the number of screws can be written as:

$$\varepsilon_{12}(\mathbf{k}) = \frac{\mathbf{V}_{w2} - \mathbf{V}_{screw} \cdot \mathbf{k}}{\mathbf{V}_{w2}},$$

where k=1,2...53 is the number of screws installed in the mixer.

1.2 Methods of experimentation

Components to be mixed are first put in the feeder. The insertions are removed and the flow starts induced by gravity forces. In order to investigate mixture quality, insertions are placed in the collector and the mass of each component is measured in each section. The photograph of the "Lab-made" static mixer is presented in figure II-2.

2 SULZER STATIC MIXER

2.1 The mixer concept

Another subject of experimental work was the mixer SULZER chemtech 4571 (DN25/33, 7 DIN2633 PN16 CH106196). This mixer is applied in powder technology to mix powders, suspensions, and also fluids.

The sketch of the mixer is shown in figure II-3. The base of the apparatus is a pipe of 27 mm in diameter. The actual diameter of the flow space is 20 mm. Static mixer elements are placed into the tube and their number can be varied from 1 to 6. The sketch of an insertion is depicted in figure II-4. The static mixer elements are placed in the mixer one after another and each element is turned 90° to the previous one. The general flow direction is illustrated in figure II-4.

To examine mixer parameters and quality of the final product, the feed and collector were made in the same way as for the "Lab-made" mixer. The feeder has been constructed from a plexiglas pipe of 20 mm diameter with cuts for removing or placing insertions (see figure II-5a). The equal volumes of the feeder tube have been marked as sections to load components with certain portions. A sketch of the insertion is shown in figure II-5b. The collector has been also made from a plexiglas pipe with the same diameter but with the length of 205 mm. The pipe is separated into eight sections and insertions can be put in and out to measure the mass of each component in sections. The cut depth is 10mm and the cuts are situated 20mm from each other (see figure II-5c).

The volume of one section in the feeder as well as in the absorber is equal to

 $V_{s1} = \pi \cdot r^2 \cdot h = \pi \cdot 10^2 \cdot 20 = 6.28 \cdot 10^3 \text{ mm}^3$

It is necessary to make analysis of the space or free volume in the mixer as a function of the number of insertions inside. The whole volume of the empty Sulzer mixer is

 $V_{w1} = \pi \cdot r^2 \cdot h = \pi \cdot 15^2 \cdot 165 = 116.572 \cdot 10^3 \text{ mm}^3$

The volume of one static mixer element was calculated from an experiment. One static mixer element was put down into a vessel entirely filled with water. Then the mass of the split water was measured. This mass was 5.09 g. The volume of one static mixer element is represented as





Figure II-3: The scheme of the SULZER mixer.





Figure II-4: The sketch of a static mixer element.



Figure II-5: Additional equipment: a – pipe feeder; b – insertion; c – pipe collector.

$$V_{\text{ins}} = \frac{m_{\text{w}}}{\rho} = \frac{5,094 \cdot 10^{-3}}{10^3} = 5.09 \cdot 10^3 \,\text{mm}^3$$

where $\,m_{\!\scriptscriptstyle \rm W}\,$ - the mass of split water

 ρ - water density.

k=1,2...6 is the number of static mixer elements installed in the mixer.

Free volume of the Sulzer mixer as a function of the number of static mixer elements



Figure II-6: Static mixer elements of the Sulzer chemtech mixer.

is represented by the following equation:

$$\varepsilon_{11}(k) = \frac{V_{w1} - V_{ins} \cdot k}{V_{w1}}$$

2.2 Methods of experimentation

The components are put in the feeder in each section with desirable contents, then the material flows through the mixer. After the mixture is in the collector, concentration of a component in each sample is easy to examine by placing the insertions and weighing the masses.

Photographs of static mixer elements and the experimental installation of the Sulzer mixer are shown in figures II-6, II-7 respectively.

<u>3 COMPARING VOLUMES OF THE MIXERS</u>

The volume difference of the mixers could be found as

$$Div\% = \frac{V_{s1} - V_{s2}}{V_{s2}} = \frac{6.6 \cdot 10^3 - 6.28 \cdot 10^3}{6.6 \cdot 10^3} \cdot 100\% = 4.85\%$$

It is clear to see that the volume diversity is not more than 5% so that it is possible to compare these mixers.

In oder to compare the volume of mixers, it is essential to calculate it in relative units. The following equation is used:



Figure II-7: Experimental installation of the Sulzer mixer (a – the mixer, b – unloading container).

$$V_\mathrm{r}\% = \frac{V_\mathrm{l}}{V_\mathrm{w}} \cdot 100\% \ ,$$

To calculate the relative number of static mixer elements the following expression is employed:

$$k_{\rm r} \% = \frac{k}{k_{\rm total}} \cdot 100\%$$
 ,

where $k_{\rm r}$ is the relative number of static mixer elements,

 $k_{\mbox{\tiny total}}$ is the total number of static mixer elements,

 \boldsymbol{k} is the number of static mixer elements installed in the mixer.

Figure II-8 shows how mixer porosity depends on the number of static mixer elements installed in the mixing zone. This is very important correlation while interpreting the mixing results because it helps us to analyse in what regime the mixer works. However, mixer porosity is not the only factor that influences mixing. The shape and the size of the static mixer elements also affect the process. Thus, if



Figure II-8: Internal porosity volume of the "Lab-made mixer and the Sulzer static mixer as a function of their relative filling.

certain number of static mixer elements is installed in the mixers so that their porosities are equal, then having the same conditions, the mixing results will not be necessarily the same. As a result, it would give us an opportunity at least to say if the regime of the mixers is the same or different under these conditions. If to compare these two mixers, it is clear the Sulzer mixer has a higher porosity range than the "Lab-made" mixer which is more likely to work at free-flow. Thus, adding mixing elements, the Sulzer mixer is expected to change the operation regime whereas the "Lab-made" mixer should work within one regime.

Operational regimes of static mixers were investigated in [34]. Three flow regimes are represented in figure II-9 according to the way of loading and discharging. The first flow regime can be characterized by dense particle bed with unlimited feed and controlled output. There is almost no free space inside the mixer and at the outlet. The second flow regime occurs in dense particle bed with unlimited feed and output. There is more free space at the inlet of the mixing zone but there is at the outlet. The third flow regime happens in loose particle bed when there is plenty of space between the particles at the mixer input and output and inside the mixing zone. This regime occurs when the inflow is controlled and the outflow is unlimited. This regime will be considered in this thesis.

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Figure II-9: Operational regimes of the static mixer according to the type of flow.

4 SYSMIX ALTERNATELY REVOLVING STATIC MIXER

4.1 The mixer concept

In order to check the models developed, experimental data on the alternately revolving static mixer SysMix are used. The experiments were done in Veszprem, Hungary and their results as well as the results of simulation have been published in [29, 30, 32]. The mixer is schematically represented in Figure II-10. It consists of two cylindrical containers of 0.3 m height and 0.3 m inner diameter (about 0.02m³ nominal volume each) and the mixing zone between them. The latter has the same dimensions as containers. Three mixing grids were arranged along the cross-section of the mixing zone, each one being composed of a layer of twisted static mixer elements (see figure II-11).

Two different characteristic sizes have been used for the static mixer: one type of them has 32 mm width (inlet and output edges) and another one has 50 mm width. The height of the individual mixer elements is generally equal to its width.



Figure II-10: Scheme of the SysMix mixer.

1 – containers for the bulk solids to be mixed; 2 – mixing zone with the grids of static mixer elements; 3 – the rotation shaft; 4 – twisted (helical) static mixer elements; 5 – bulk solid layers into the lower container.



Figure II-11: Arrangement of the static mixer elements in the grids.

4.2 Methods of experimentation

During operation, the mixer was alternately revolved around the horizontal shaft at the middle height of the mixing zone. At first, one of the containers was filled with the bulk solids to be mixed and this container was joined to the mixing section. Then the mixer was turned by 180° and stopped. During revolution, the solid material moved up together with the container (see figure II-12). After being stopped, material flowed down through the mixing zone into the other container. The components change their places as it is shown in figure II-12. Then the mixer was turned again by

180° but in the opposite direction. This process was periodically repeated during the experimental time. The time of a 180° rotation was 1.5 second followed by a standstill of 3.0 seconds. Unfortunately, there are no published data on the mixer porosity.



Figure II-12: A sketch of mixture revolution (a - arrangements of components before revolution, b - arrangements of components after revolution).

The operation principle of this mixer is similar to classical tumbler mixers but there are some differences. One is that the rotation of the mixer is not continuous, i.e. a rotation period is always followed by a standstill. The second difference is that the direction of the rotation is reversed after every 180° turn. The last difference is that during the motion of a tumbler mixer there is a relative displacement of particles (e.g. cascade movement).

5 MATERIALS

Density of the components was measured by Pycnometre (ACCU PYC1330), Micrometrics. This device measures exact volume occupied with material in a part of a well known volume. Helium is introduced into the volume and then real density of material is calculated. Each value of a volume is an average result of 25 experiments. These measurements were done with the volume of 10 cm³. The list of the components used in this work is given in table II-1.

Material	Particle density (kg/m ³)
SiC	3130
Al ₂ O ₃ aluminum oxide	3903
sugar	1559
semolina	1470
couscous	1440
millet	1410
quartz sand	2650
sodium chloride	2160
wheat flour	1510
polypropylene granules	910

Table II-1: Density of the components used in the experiments.

The particle size distribution experiments were carried out according to the following principle. A portion of a certain component was taken and its total mass was measured. Afterwards the material passed trough the sieves of different diameters and the mass of particles passed through the sieves was measured. The results were put into a table (see Appendix). Passage of material with the size x_i through the sieves was calculated as

$$Q_i \% = \frac{M_i}{M_{\text{total}}} \cdot 100\%$$
(II-1)

where M_i is the mass of material of the x^{th} size passed through the sieves; M_{total} is the total mass of the material examined.

It is necessary to calculate the relative difference in particle sizes using the following equation:

$$s_{pan} = \frac{d_{90} - d_{10}}{d_{50}} \tag{II-2}$$

where d_{10} is the accepted minimal diameter of particles; d_{90} is the accepted maximal diameter of particles; d_{50} is the average diameter of particles. SPAN is the relative diversity of particle sizes with regard to d_{50} . The results of calculation are given in table II-2.

Material	d ₁₀ ,microns	d ₉₀ ,microns	d ₅₀ , microns	SPAN
Millet	1700	2400	1900	0,368
SiC	400	640	560	0,429
Corindon	1050	1550	1230	0,407
Sugar	530	780	680	0,368
Couscous	1020	1550	1180	0,449
Semolina	200	480	330	0,848

Table II-2: Average size of particles.

The differences in diameters of components are fairly seen. Using different pairs of materials can help investigate how static mixers operate with components differed with the density and particle size distribution. Table II-2 also shows the average size of particles used for the experiments.

CHAPTER III:

ONE AND TWO DIMENSIONAL MARKOV CHAIN MODELS FOR STATIC MIXERS

1 ONE DIMENSIONAL MARKOV CHAIN MODELS FOR STATIC MIXERS

Only one dimensional representation of the models has been used for static mixers by different authors, e.g., Gyenis [25, 26, 27]. Fan [36, 47] also suggested a one dimensional approach to mixing simulation of static mixers on the basis of the theory of Markov chains. The 1D approach allows modeling flow in one direction: axial or crosswise, what can be considered as a limitation of the model. Very often mixing occurs in several directions and, thus 2D models should be used to describe the process. But from the point of view of model development, a better strategy is to build a 2D model on the basis of a 1D model. This is the reason why we will first focus on the 1D chain models.

1.1 Model description. Algorithms of its numerical representation

1.1.1 A model scheme

Suppose that there is a vertical tube with two components A and B at the top of it (see figure III-1). At the very initial state, A and B are completely separated. Let us divide the total volume of A and B into, say, 10 sections (samples): 5 for A, and 5 for B. In reality, it may be modified if the scale of sampling has to be smaller while estimating quality. Therefore, the volume of a section will be the scale of scrutiny in this case. It is necessary to note that the total volume of A and B in the tube and volume of the components in each cell are constant. Thus, the volume of one section is the scale of sampling in our case.

The central part of the tube is a mixing zone, no matter of what kind. There may be helical elements in it, or even nothing – just a space for free fall. Let us make the materials move downward. After the process is ended, the material is collected at the bottom of the tube and it occupies the same volume as at the top of the tube. Each sample (or some of them) contains both components A and B in the absorber. Thus all the material is represented by its mixture at the lower part of the tube.



Figure III-1: The general scheme of the process and its chain model.

1.1.2 The matrix of transition probabilities

In order to build the matrix of transition probabilities for the mixing zone, let us present it as a sequence of perfectly mixed cells. These cells can correspond to mixing elements inserted into the mixing zone, or can be chosen arbitrary to define the zone as several spatial sections. A time step Δt is to be chosen small enough to as-

sure that particles can transit no further but to a neighboring cell. Under these assumptions the matrix of transition probabilities has the form:

$$\mathbf{M} = \begin{bmatrix} \mathbf{p}_{s} + \mathbf{p}_{u} & \mathbf{p}_{u} & 0 & 0 & 0 & 0 \\ \mathbf{p}_{d} & \mathbf{p}_{s} & \mathbf{p}_{u} & 0 & 0 & 0 \\ 0 & \mathbf{p}_{d} & \mathbf{p}_{s} & \mathbf{p}_{u} & 0 & 0 \\ 0 & 0 & \mathbf{p}_{d} & \mathbf{p}_{s} & \mathbf{p}_{u} & 0 \\ 0 & 0 & 0 & \mathbf{p}_{d} & \mathbf{p}_{s} & 0 \\ 0 & 0 & 0 & 0 & \mathbf{p}_{d} & \mathbf{p}_{abs} \end{bmatrix}$$

(III -1)

where p_u , p_d and p_s are probabilities of material to go backward, forward and to remain in a cell during the period of time Δt which is considered to be equal to the duration of one portion entering into the mixing zone; p_{abs} is the probability of material to stay in the particle absorber cell that follows the mixing zone (if $p_{abs}=1$, what corresponds to the reality, it is the state in the last cell of the mixing zone, i.e., on the bottom of the mixer. This state describes accumulation of material; if $p_{abs}=0$ then this state is actually the outflow into the absorber at every transition, what can be used to draw residence time distribution easier. Every column of the matrix should meet the condition of normalization (chapter 1, (I-17, I-18)).

Considering that the tight motion of free falling particles is impossible in the opposite upward direction, $p_u=0$ and thus $p_d =1$ - p_s . This means that the matrix of transition probabilities is defined by a single parameter p_s , which however might be different for different components of the mixture. It should be mentioned that components A and B, may significantly differ in physical properties as density, size and shape what will make each component flow through the mixing zone with different velocity. In order to take this effect into consideration let us represent the process in the form of two Markov chains (the first chain describes motion of the component A and the second chain is for the component B). As for the model structure, the two chains for models seem to be independent, however implicitly the motion of one component in the media of the other one is taken into account while identifying the model parameters p_A and p_B . For example, if the component A is the same as it was before and the component B is substituted by another one with different physical properties, the value of p_B will be different and the value of p_A will be also different although the

although the component A is the same. It is necessary to note one point that the results of simulation in these two chains influence each other once more while analyzing the micro outflows of each component into the absorbing state at every transition and recalculating them in samples (this procedure will be explained below). That is why the mixing process does not run independently in these two chains.

Therefore, as soon as we use the Eulerian approach we are interested in how many particles of each component are in a cell and how this situation changes after every transition. According to the information mentioned above, the matrices of transition probabilities for the components A and B will take form:

$$\mathbf{M}_{A} = \begin{bmatrix} 1 - p_{A} & 0 & \dots & 0 & 0 \\ p_{A} & 1 - p_{A} & \dots & 0 & 0 \\ 0 & p_{A} & \dots & 0 & 0 \\ \dots & \dots & \dots & \dots & \dots \\ 0 & 0 & \dots & p_{A} & 1 \end{bmatrix}$$
(III -2)
$$\mathbf{M}_{B} = \begin{bmatrix} 1 - p_{B} & 0 & \dots & 0 & 0 \\ p_{B} & 1 - p_{B} & \dots & 0 & 0 \\ 0 & p_{B} & \dots & 0 & 0 \\ \dots & \dots & \dots & \dots & \dots \\ 0 & 0 & \dots & p_{B} & 1 \end{bmatrix}$$
(III-3)

where p_A , p_B are probabilities of the components A and B to move downward during the period of time Δt , which is equal to the duration of one portion entering into the mixing zone. Matrices M_A and M_B are the matrices of transition probabilities for the components A and B respectively.

If these matrices \mathbf{M} are constant (homogeneous Markov chain process) the kinetics of material motion through the mixing zone is defined by the following matrix equation:

$$S_{A}^{i+1} = M_{A} (S_{A}^{i} + S_{fA}^{i})$$
 (III-4)

$$\mathbf{S}_{\mathsf{B}}^{\mathsf{i}+1} = \mathbf{M}_{\mathsf{B}} \left(\mathbf{S}_{\mathsf{B}}^{\mathsf{i}} + \mathbf{S}_{\mathsf{f}\mathsf{B}}^{\mathsf{i}} \right) \tag{III-5}$$

$$\mathbf{S}_{A} = \begin{cases} \mathbf{S}_{A1} \\ \mathbf{S}_{A2} \\ \mathbf{S}_{A3} \\ \dots \\ \mathbf{S}_{An} \end{cases}, \qquad \mathbf{S}_{B} = \begin{cases} \mathbf{S}_{B1} \\ \mathbf{S}_{B2} \\ \mathbf{S}_{B3} \\ \dots \\ \mathbf{S}_{Bn} \end{cases}$$
(III-6)

where S_{A} , S_{B} are state column vectors of the components A and B respectively in the mixing zone, $S_{fA} S_{fB}$ are column vectors of material feeding into the mixing zone.

Apparently during the first material passage through the mixing zone, the pure component A will exit first and then the mixture of the components A and B will follow due to the delay in the mixing zone.

1.1.3 Transformation of microstates to macrostates

As it was mentioned above the matrices M_A and M_B are different in general case but according to the accepted assumptions they are:

1) constant with time (homogeneous chain);

2) state independent, i.e., the matrices do not depend on concentration of A and B at the bottom (linear chain).

The last point needs some comments. Suppose A is steel balls and B is couscous. A is falling obviously faster, and falls of both ones are more or less independent. However, if A is couscous, and B is steel balls, the steel balls have to move through couscous. In this case the steel balls have additional resistance to their mo-



tion, which depends on concentration of couscous in a cell. This phenomenon is ignored at the moment. The components are approaching the bottom of the tube according to the RTD histogram for each component (see figure III-2).

Figure III-2: RTD histogram.

However the point is that material flows out of the mixing zone at every time instant Δt in small quantities

which are not equal to the scale of scrutiny (sample size), according to RTD. We will call a sample entirely occupied by material as a macrostate and the quantity of material leaving the mixing zone at the time Δt as a microstate. Thus, every micro state is a RTD value at certain time instant. Therefore, we have samples or *macrostates at*

the mixer inlet and after the process we obtaine RTD or microstates at the mixer outlet. An example of outflow after one passage in the form of microstates is shown in figure III–3(a), where solid horizontal lines correspond to macrostates and hatch lines correspond to macrostates. It is clear to see that they do not coincide with each other, i.e. there are no cells or samples where concentration can be determined.



Figure III-3: Distribution of the component A at the bottom after the first passage with $p_A=0,7$ and $p_B=0,7$; a) in microstates, b) in macrostates.

If we like to know what happens with the mixture after the second transition, it is necessary to put the obtained mixture at the mixer inlet. However, we can not put the RTD at the mixer inlet due to two reasons: firstly, the size of the vector with RTD data will grow rapidly after every passage of material what makes the procedure impossible to calculate and secondly, it is necessary to have samples where process characteristics as concentration and variance can be found. Consequently, the microstates or RTD shoud be transformed to macrostates.

The procedure of recollection can be understood from the graphs in figure III-4. Putting p_{abs} =1, the last state S_m^{i} becomes a cumulative outflow at the bottom. The cumulative outflow of mixture A+B, and cumulative outflows of the components A and B separately are calculated after every transition. Ordinate axis is divided into

the intervals (samples or macrostates) which are considered at the mixer inlet. When the cumulative sum of A+B becomes equal to a macrostate, we look at how much of each component is in the macrostate using the other two curves. Afterwards a macrostate or a sample is formed and concentration of the components is calculated.



Figure III-4: Cumulative collection of the component A and the mixture at the bottom.

Some additional simulations are shown in figure III-5. The stripes on the left graph show the microstates of the mixture at the bottom after the first passage. Edges of the stripes do not coincide with the levels corresponding to the macro states. The graph on the right shows the same distribution after recollection. Every macro state contains only two stripes, and the relative width of every strip is the relative concentration of the component in the macro state.

Thus, the obtained state vector that

a) characterizes the state of the mixture in terms of macro states, which can be measured;

b) becomes the initial feed vector for modeling the next passage being turned upside down.

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Figure III-5: Evolution of the initial state vector during the first passage.

1.1.4 Realization of mixer feeding in the model

After the first revolution of the mixer, the state vector at the mixer bottom is reversed by 180° and finally it becomes the initial feeding state vector. This vector is represented by macrostates. The scheme of the material injection from the feeder (or uploading container) into the mixing zone is proposed on figure III-6. As long as the material injection occurs into the first cell of the mixing zone, the following equation can be written:

$$S_{af1}=u(i) - u(i - 6), S_{bf1}=u(i - 6) - u(i - 11),$$
 (III-7)

where $u(i - i_0)$ is the unit jump function. All other elements of the feeding vector are equal to zero. Firstly, material enters the mixing zone (into the first cell of the chain) from the last 10^{th} feeder section. Afterwards, the material goes from the 9^{th} section to the 10^{th} section, then from the 8^{th} to the 9^{th} and so on after every transition. Let us suppose that material is moved down on one section after one transition. Figure III– 6a gives the initial distribution of material in the uploading container (feeder). Figure III-6b and III-6c show the initial distribution after one and two transitions respectively. It is assumed that the components do not mix during their motion through the feeder (plug flow).



Figure III–6: The scheme of material injection from the feeder into the mixing zone, i.e. into the first cell of the Markov chain (a – initial distribution, b - after 1^{st} transition, c - after 2^{nd} transition).

1.1.5 Consecutive and reversed material loading of static mixers

After each revolution, the distribution of components along the whole volume will change aiming to become more homogeneous. Some mixing cycles are shown in figure III-7 in which mixture is reversed after each passage. But mixture can be transmitted to the mixer inlet without being changed, i.e., with the same order as it was at the mixer bottom during the previous mixing cycle. This case can be seen if two static mixers are installed one after another or if the mixing zone is lengthened up and the number of static mixer elements inside is raised up (see figure III-8).

The principle of material feeding will be regarded in details below. By repeating calculations, it is possible to approach asymptotical mixture homogeneity and determine the number of mixer rotations providing necessary mixture quality (say, coefficient of variation CV=6% that is generally used in industry). CHAPTER III: ONE AND TWO DIMENSIONAL MODELS FOR STATIC MIXERS



Figure III-7: Mixing with mixture reverse for the next mixing cycle (static laboratory mixer).



Figure III-8: Mixing without mixture reverse for the next mixing cycle. This case can be seen if two static mixers are installed one after another or if the mixing zone is lengthened up (static mixer SULZER).

1.2 Evolution of the mixture state and its numerical characteristics

The key operator of the mixture evolution is the matrix of transition probabilities. As it was mentioned above, the probabilities situated below the main diagonal are responsible for the portions of material going down. The probabilities situated on the main diagonal are responsible for the portions of material staying in corresponding cells (the probabilities p_A , p_B are determined for the case of empty mixing zone, i.e. without mixing elements). The size of the matrix can be chosen according to the number of mixing elements. The last row and column define the accumulative cell, i.e., the absorber or the cell into which material goes.

As far as probabilities are determined for the mixing zone without mixing elements, we have to think of how to take them into account by the model when some of them are installed. For this, we will use coefficients of delay α , which takes into account the effect of a mixing element. Each probability of the matrix is multiplied by this coefficient and if there are no mixing elements in the zone, the value of all the coefficients is equal to 1. If some mixing elements are installed, the delay coefficients which stay in the columns corresponding to the position of the elements will be different to 1. The other delay coefficients will be still equal to 1. It is illustrated on a simple example in figure III-9. The mixing zone consists of 4 cells for mixing elements and the fifth one is for the absorbing cell. If one mixing element is installed in the first cell of the mixing zone, the coefficient α_1 staying in the first column (this column corresponds to the 1st cell of the zone) will be equal to some value, say, 0.8 (see figure III-9a). Identification of this parameter will be discussed later. The other coefficients α will be equal to 1. Example on figure III-9b is given for the case when two mixing



Figure III–9: Values of coefficients of delay depending on the number of mixing elements in the mixing zone (a – one mixing element is in the 1^{st} cell of the mixing zone; b – two mixing elements are in the 2^{nd} and 3^{rd} cells of the mixing zone).

elements are installed in the 2nd and 3rd cells. Consequently, α_2 and α_3 staying in the 2nd and 3rd columns will be equal to 0.8, the others will be equal to 1. It must be remarked, that the mixing elements were assumed to cause equal delay.
The probabilities p_A , p_B are determined from experiments. State vectors determine concentration distribution of the components. Let us assume that mixing of components with identical physical properties is run. It is obvious that, if matrices III-3, III-4 are the same for both components, their distribution will tend asymptotically to homogeneous one and the question is how many revolutions of the mixer needs to be made to guarantee the mixture quality. It is worth mentioning that the number of transitions during one passage of material through the mixing zone is not equal to the number of sections or macrostates. It happens because the outflow goes in microstates. The number of transitions must be so that material has entirely left the mixing zone. In other words, material should be given time to flow out of the mixer.

Some examples of simulation are given in figure III-10. Evolution of the state vector is shown for identical matrices M_A and M_B with $p_A=p_B=0.7$. The results are represented in the form of recollected distribution (macro states) at the bottom after different number of passages. After 32 rotations of the mixer, the distribution approaches homogeneity for the identical matrices M_A and M_B .

Another example is shown in figure III-11 for different matrices M_A and M_B with $p_A=0.6$ and $p_B=0.8$. The concentration profile is not even after 32 rotations here, i.e., what shows that mixing is worse than in the previous case (figure III-10).

Figure III-12 shows the evolution of the mixture homogeneity presented by the variance of the component A at the bottom in macro states. The evolution behavior strongly differs for identical and different transition matrices. The variance monotonously decreases for identical matrices, and it is asymptotically equal to zero.

In the case of different matrices, a most important fact is that the distribution can be more homogeneous with certain number of mixer revolutions (e.g. $\sigma_A^2=0.08$ at 13 passages) rather than with higher number of revolutions (e.g. $\sigma_A^2=0.12$ at 40 passages). This effect can be caused by segregation due to different properties of components and consequently different values of transition probabilities.

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Figure III-10: Distribution of components at the bottom after different number of passages $(p_A=p_B=0.7)$.







1.3 Results of mathematical simulation. Optimal number of revolutions

1.3.1 Reversed loading

Let us consider an alternately revolving static mixer with 6 mixing elements that provide higher mixing intensity. Then the process is defined by the matrices of transition probabilities with size 7×7 so that each mixing element corresponds to one element of the matrices. The initial state vectors will be S_A = [0 0 0 0 0 1 1 1 1 1] and S_B = [1 1 1 1 1 0 0 0 0 0]. If the components have the same physical properties, the probabilities p_A and p_B are equal to each other. Figure III-13 shows concentration distribution of the component A along sections with different number of material passages through the mixer for p_A =0.8 and p_B =0.8. Having such probability values, the homogeneity is reached after 9 mixer revolutions. The concentration distribution becomes more homogeneous after every mixer revolution, since the components have the same physical properties. Segregation or any worse homogeneity is not observed with increasing passage number. In this case the question is how many mixer revolutions should be made to reach the quality required (e.g. 6% of CV). Variance curve is given on figure III-14 for the same state vector but different probabilities and different



N, section number

Figure III-13: Concentration distribution in sections for $p_A=p_B=0.8$, 10 mixing elements and different number of passages.



Figure III-14: Variance versus the number of passages for different probabilities p_A and p_B , 10 mixing elements.



Figure III-15: The necessary number of transitions for reaching CV=6% versus probability value ($p_A=p_B$).

number of static mixing elements. The less the probabilities p_A and p_B are, the more intensive the process will be, what means that the variance will be diminishing quicker. Figure III-15 shows the necessary number of transitions for achieving CV=6% for different values of transition probabilities ($p_A=p_B$). It is clear to see that when the probability value is close to 1, the process is very sensitive to

its change and little variation causes significant change in the process. The further the probability value from 1 is, the less sensitive the process becomes. Let us regard a situation when the components are significantly different in their physical properties, i.e., they tend to segregate. Let us put $p_A=0.9$, and change the probability p_B from 0.9 to 0.3. As a result, the component B will be falling down with lower probability than the component A. The initial state vectors are the same as for the previous case. The variance curve, corresponding to this case, is shown in figure III-16. The example shows that with an odd number of material passages through the mixing zone, variance has values several times lower than if the number of passages is



Figure III-16: Variance versus the number of passages for p_A =0.9 and different values of p_B (a case of revolving static mixer).

even. In this case the component, having higher probability was initially placed at the top of the container. Hence, mixing is worth stopping after an odd number of passages. However, CV=6% is not achieved in this case, except if the transition probabilities are equal (if $p_A=p_B=0.9$, CV=6% is reached after 6 passages). Moreover, having certain quality requirements, it might be more reasonable to stop the process even after the first passage. If the difference in probabilities is higher, variance decreases faster but it has asymptotically higher value. Figure III-17 shows concentration distribution along sections of material collector. It is clear to see that mixture is more homogeneous in the middle sections and less homogeneous at the edges.



Figure III-17: The concentration distribution in sections for different probabilities after the10th material passage through the mixer (a case of alternately revolving static mixer).

Let us take an example for which the component, having higher downward probability value is placed at the bottom of the loading container. Variance as well as concentration distribution in sections after the 10th passage are shown in figures III-18 and III-19. The process behavior has not changed except that a more homogeneous state is reached after an even number of material passages, so that the process should be stopped at these points. Comparing the variance values, we conclude that, in the first case, mixture quality is approximately the same after one passage as for the second case after two passages (if $p_A=0.9$, $p_B=0.6 \sigma_A^2 = 0.038$). Thus, the rea-

sonable position of the component, having higher downward probability is at the top of the feeder. Afterwards it is better to unload the mixer after odd number of material passages.



Figure III-19: The concentration distribution in sections with different probabilities after the10th material passage through the mixer (a case of alternately revolving static mixer).

1.3.2 Consecutive loading

The next example is taken for the Sulzer static mixer. The continuous feeding is possible to organize with this model but different experiments would be necessary to make. If we suppose that continuous feeding is a series of feedings acting within a time interval, we can thus focus on the feed acting within time interval. If that is the case, the model will differ from the previous one, only by the method of material feed-ing. Here, the mixture is not reversed after every passage. It goes to the next processing stage in the same order. Let us introduce the state vectors S_{A} = [0 0 0 0 0 1 1 1 1] and S_{B} = [1 1 1 1 1 0 0 0 0 0]. The component having higher probability value will then precede the component having lower probability value at the mixer inlet. The principle of model developing of such a mixer is similar to the previous one regarded and mixture goes to the next processing stage in the same order as it was collected at the mixer outlet. Obviously, variance can not be oscillating in this case (see figures III-20 and III-21). CV=6% is achieved only if the probabilities p_A and p_B are equal. Here, this value is reached after 15 passages for $p_A = p_B = 0.95$.



Figure III-20: Variance versus the number of passages for p_A =0.95 and different values of p_B (a case of non revolving static mixer).



Figure III-21: The concentration distribution in sections with different probabilities after the10th material passage through the mixer (a case of non revolving static mixer).

Similar example is shown in figures III-22 and III-23. Here, loading is also consecutive from one passage to another but initial distribution of the components is a bit different. The component having higher probability value initially follows the other one moving with lower probability. Accordingly, initial state vectors are S_{A} = [1 1 1 1 1 0 0 0 0 0] and S_{B} = [1 1 1 1 1 0 0 0 0 0]. Variance curve in figure III-22 has a minimum after the 5th passage for p_{A} =0.95, p_{B} =0.9, i.e., if to increase the number of material



Figure III-22: Variance versus the number of passages for $p_B=0.95$ and different values of p_A (a case of non revolving static mixer).

passages through the mixer, mixture will become less homogeneous due to segregation. Therefore, it is worthwhile to choose a time moment when mixing should be stopped and the mixture should be unloaded or conveyed to the next processing stage. If that is the case, it is more reasonable to put initially the component having higher probability value on the top of the other component. The CV=6% is not achieved in this case except if the transition probabilities are equal.



Figure III-23: The concentration distribution in sections with different probabilities after the10th material passage through the mixer (a case of non revolving static mixer).

1.4 Choice of the mixer



1.4.1 Mixing time definition

Accurate calculation of the mixing time is a very difficult problem. Hence, let us just specify time instants when particles are falling through the zones of the mixer (see figure III-24).

The total time of one passage can be found using the following equation:

$$\mathbf{t}_{\text{passage}} = \mathbf{t}_1 + \mathbf{t}_2 , \qquad (III-8)$$

Figure III-24: Zones of particle passage in the mixer.

where t_1 is the time of particle motion along the loading container till the first row of mixing elements; t_2 is the time of particle motion through the mixing zone. The total time of material handling can be described as

$$t_{\text{handling}} = (t_1 + t_2) \cdot k + t_{\text{loading / unloading}}$$
(III-9)

where k is the number of mixer rotations; $t_{loading/unloading}$ is the time of mixer loading and unloading, (the time of rotation is included).

1.4.2 Comparison of reversed and consecutive loading in a static mixer

Mixture is reversed in the alternately revolving static mixer after each cycle. In this case, the material came later and been at the top of the unloading container, will go first to the mixing zone at the next passage. However, it seems that it is just possible to add some more mixing elements and the same mixing effect seems to be obtained without any mixer rotation. In order to investigate this particularity let us regard some graphs with flipping over the state vector turn and without it.

We will take the "Lab-made" mixer for investigation. If the probabilities are equal i.e., $p_A=p_B=$ 0.92, and the number of mixing sets is N=2, variance monotonously diminishes (see figure III-25). Variance curves are given firstly for the case of



Figure III-25: Variance versus the number of mixture passages with and without mixture turn ($p_A=p_B=0.92$, N=2).

mixture turn done every next mixing cycle and secondly for the case when mixture is introduced into the mixer in the same order (i.e., it may correspond to the case of lengthening mixing zone and adding some extra mixing elements). The curves almost coincide.

Here, the choice of consecutive fed mixer with long mixing zone or revolving mixer with a short mixing zone is determined by the installation requirements in the technological process. If there is enough place, using mixer with long mixing zone would be more reasonable as the mixing cycle time will be less. If that is so, it is possible to design the mixing zone long enough to avoid additional material passage through the mixer.

The variance curves for the case of the probabilities $p_A=0.88 p_B=0.92$, and the number of mixing sets N=2 are represented in figure III-26. Here, one of the curves corresponds to consecutive loading of material. The other curve corresponds to loading of material when mixture is reversed before every new mixing cycle (SysMix mixer).

Now the component, having higher downward probability is initially placed at the top of the upper loading container. The variance curve of non-revolving process borders upon the axes and it has a minimum at the point of 4 material passages. The variance value is $\sigma_A^2 = 0.002$ at this point and $\sigma_A^2 = 0.012$ for the case of revolving



Figure III-26: Variance versus the number of mixture passages with mixture turn and without it (p_A =0.88 p_B =0.92, N=2).

process. Thus, it can be stated that non-revolving static mixer would be more reasonable to use. However, the process should be stopped after 4 passages of material since further mixing will result in mixture segregation. If there is difficulty in stopping process at the point of the variance minimum, alternately revolving static mixer can be used. Here, the variance curve is monotonously oscillating and it has a value of $\sigma_A^2 = 0.002$ at the point of 8 material passages. Thus, it is possible to reach better mixture quality for the revolving process but with longer mixing time.

The variance curves for consecutive and reversed loading for the probabilities $p_B=0.88 p_A=0.92$, and the number of mixing sets N=2 are represented in figure III-27. The component, having higher downward probability, is placed at the bottom of the upper loading container. Examining the curve of consecutive loading, the variance monotonously diminishes and reaches the value $\sigma_A^2 = 0.042$ at the point of 10 passages, what says about low mixture quality. With reversed loading, variance diminishes faster and it has lower value $\sigma_A^2 = 0.0003$ at the point of 10 passages. Variance has oscillating character with odd and even number of passages and oscillations become insignificant with the passage rise. Thus it is more reasonable to use alternately revolving static mixer with this initial distribution of components.

If to compare figures III-26 and III-27, say, a point after 1 passage, we can state that it is better to place initially the component having higher downward prob-



Figure III-27: Variance versus the number of mixture passages with and without mixture turn ($p_A=0.92 p_B=0.88$, N=2).

ability at the top of the container especially if consecutive loading is used $(\sigma_A^2 = 0.075 \text{ and } \sigma_A^2 = 0.13 \text{ when the component having high probability is placed at the top and at the bottom consequently).$

1.5 Length of the loading container

In order to investigate how mixing time changes with variation of mixer loading, let us make the following numerical experiment using the model. The size of the feed vector corresponds to the number of sections in the loading container. Hence, if to vary the number of its elements, it is possible to simulate the variation of mixer loading. The more material is in the loading container, the more sections are to be considered and consequently the more elements the state feed vector has. Here is an example in which state feed vectors contain the relative composition of the components A and B as [2, 3], [4, 6], [8, 12], [16, 24], [32, 48]. The first number in the square brackets means that 2 sections of the upper loading container are filled with the component A and 3 sections with the component B and so on. Therefore, we will consider the number of elements of the state feed vectors as 5, 10, 20, 40 and 80. Kinetics of variance distribution as a function of the number of passages is shown in figure III-28 for different number of elements in the state feed vector ($p_A=p_B=0.92$,





N=10). The longer the length of the sate feed vector is (mass of material is increased), the more mixing passages are required to reach the same value of variance.

Figure III-28 allows finding the number of transitions required to reach the same value of variance for each number of sections in the loading container (see figure III-29). The less the variance value is, the more transitions is necessary to reach it. For example, if the number of sections is 40, then we will need 8 transitions to reach CV=84,9%, 18 transitions to reach CV=74,8% and 32 transitions to reach CV=63,3%. The curves become more linear in the area of high number of sections.



Figure III-29: The number of transitions required for reaching variance values given versus number of sections in the loading container.

These curves give us a lot of information, however every time when the number of sections in the loading container is different, the mass of components will be different as well. Therefore we will obtain different mass of final product what makes it more difficult to compare the results. In order to make the results more comparable let us reformulate the requirements for the information given by the graph. We would like to know how much time is necessary to produce certain mass of mixture of the equal quality demanded as a function of the number of sections in the loading container. This total time should include the time of material handling (which is calculated as a sum of loading/unloading time, and passage time multiplied by the number of mixer revolutions) multiplied by the number of cycles to process the required mass of the final product. Therefore, the equation for its calculation takes form:

$$t_{total} = ((t_1 + t_2) \cdot k + t_{loading} / u_{nloading}) \cdot \frac{m_{req}}{m_{ph}}, \qquad (III-10)$$

where m_{req} – required mass of final product, m_{ph} – mass of mixture after one cycle (a cycle here is considered to be the process time from charging to discharging). As it is supposed that one section corresponds to certain mass, we took the number of sections in the loading container instead of pure mass.

Let us assume that the time of loading/unloading is equal to two transitions (see figure III-30a). The curves have a minimum what means that there is an optimal number of sections in the loading container that gives minimum mixing time to produce the same mass of final product with the same value of CV. For instance, the minimum time for CV=63.3% is achieved with 10 sections in the container, for CV=74.8% is achieved with 10 sections, CV=84.9% is achieved with 20 sections. Therefore, the optimal number of sections is different for different values of CV. A less number of sections is recommended for reaching low values of CV. Figure III-30b is given for the time of loading and unloading $t_{loading/unloading} = 3$ transitions.



Figure III-30: Total mixing time for preparing the same mass of mixture of the same quality versus the number of sections (a - $t_{loading/unloading} = 2$ transitions, b - $t_{loading/unloading} = 3$ transitions, c - $t_{loading/unloading} = 5$ transitions, d - $t_{loading/unloading} = 10$ transitions).

Here, we can observe similar picture as in figure III-30a. The optimal number of sections displaces to the right. Figure III-30c allows us to see this effect more distinctly. In this way, the minimum time for CV=63.3% is achieved with 20 sections, for CV=74.8% is achieved with 20 sections, CV=84.9% is achieved with 40 sections. Similar effect can be seen on figure III-30d. The value of mixing time rises with increasing the quality requirements and the time of loading and unloading, what is logical. In conclusion, the optimal number of sections should be searched and the model facilitates this process when different parameters are changing.

2 A TWO-DIMENSIONAL MODEL OF AXIAL-CROSSWISE MIXING IN ALER-NATELY REVOLVING STATIC MIXERS

2.1 Model description. Algorithms of its numerical representation

2.1.1 Model scheme

In this chapter, the one dimensional model, developed earlier, is generalized for modeling two-dimensional process, when mixing occurs both in vertical and horizontal directions. It should be also applied for two static mixers considered earlier but there is difficulty in getting the experimental results in horizontal direction in cause of small diameter of these mixers. Alternately revolving static mixer SysMix will be the prototype for building the model. Horizontal mixing is determinant in this mixer. This mixer consists of two cylinders and the mixing zone placed between them. The mixing zone has a mixing grid with horizontally placed mixing elements. The mixer can be rotated along the horizontal shaft. Mixture is therefore forced by gravity to fall down from the upper container to the lower container through the mixing zone. As soon as all material reaches the lower container, the mixer is rotated by 180° and the process repeats (see chapter II).

As far as SysMix mixer has a large diameter, we will apply a two-dimensional approach to investigate the mixture evolution in both directions. It's just because the experimental crosswise data are available. The scheme of the process for m columns is represented in figure III-31. The component A is placed in the left part of the upper container and the component B in its right part.

The model consists of three basic parts:

- 1. feed of material from the upper container to the mixing zone;
- 2. motion of material through the mixing zone;
- 3. recollection of microstates into macrostates.

2.1.2 Realization of mixer feeding in the model

Material feed is organized by means of special feeder or upper loading container. The latter is represented by the two-dimensional cell space where the number of cells is equal to the number of columns of mixing elements and the number of rows equals to the number of samples chosen in vertical direction. The component A



Figure III-31: The scheme of the process.

is placed in the upper left hand part of the feeder (upper container) and the component B is placed in its upper right hand part. The scheme of material injection into the mixing zone represented by 4 columns is shown in figure III-32. Firstly, material enters the mixing zone (the first row of the chain cells) from the last the 10^{th} row of the feeder sections. As soon as the mixer volume is represented by columns and rows of mixing cells, i.e. the cells are situated both in axial and crosswise directions, then material can move from the 10^{th} column only to the first cell of the same column in the mixing zone. The same point is true for the other columns of the feeder. Afterwards, material moves from the 9^{th} feeder row to the 10^{th} , then it goes from the 8^{th} to the 9^{th} and so on. We assume that while material is shifting and entering the mixing zone, components do not mix (plug flow). Thus, material is considered to be shifted in one section after one transition). Figure III-32a shows the state after one transition and figure III-32b shows the state after two transitions. The volume of material is constant inside the mixer.



Figure III-32: The scheme of material injection into the mixing zone, i.e. into the first row of cells.

2.1.3 Matrix of transition probabilities

Mixing zone is represented by a block matrix of transition probabilities since mixing occurs in two directions. Notwithstanding the procedure of describing main characteristics and two-dimensional model operators is given in [66] in details, let us briefly regard its application to the present process. Let us take a 3x3 two-dimensional cell model with rectangular cells as it is shown in figure III-33.

Let us suppose that we have a state probability distributed in isotropic medium which is represented by ideal cells. Consequently, the state probability distribution along cells can be defined by the matrix

$$\mathbf{S}_{m} = \begin{bmatrix} \mathbf{S}_{11} & \mathbf{S}_{12} & \mathbf{S}_{13} \\ \mathbf{S}_{21} & \mathbf{S}_{22} & \mathbf{S}_{23} \\ \mathbf{S}_{31} & \mathbf{S}_{32} & \mathbf{S}_{33} \end{bmatrix}$$
(III-11)

However, in order to build the matrix and perform next matrix operations, model cells should be numbered in direct sequence as it is shown in figure III-33 and the matrix,

$$\mathbf{S}_{m} = \begin{bmatrix} \mathbf{S}_{1} & \mathbf{S}_{4} & \mathbf{S}_{7} \\ \mathbf{S}_{2} & \mathbf{S}_{5} & \mathbf{S}_{8} \\ \mathbf{S}_{3} & \mathbf{S}_{6} & \mathbf{S}_{9} \end{bmatrix}$$
(III-12)

that should be transformed into the state vector

$$\mathbf{S} = \begin{bmatrix} \mathbf{S}_1 \\ \mathbf{S}_2 \\ \dots \\ \mathbf{S}_8 \\ \mathbf{S}_9 \end{bmatrix}$$
(III-13)

If the numeration is so, the rule of building the matrix of transition probabilities becomes obvious. The size of the matrix is 9x9. Each column contains transition probabilities from the given cell to the neighboring cells according to its direct unary nu-



Figure III-33: The scheme of the cell model of the state size 3x3.

meration. The main matrix diagonal has probabilities to remain in cells during one transition. These probabilities can be obtained by subtracting the sum of transition probabilities to the neighboring cells from 1. The probabilities of such transitions should be placed in the matrix columns as it is given below

Let us regard the mixing zone itself. It is assumed to be represented by mixing elements, axially installed in the middle of the mixer. Each element is shifted one from another in 90° (axial direction) [25]. Consequently, material motion will occur along two sides of each mixing element so that it goes to the mixer bottom and other neighboring zones. A cell model of the process is represented in figure III-34. Each cell represents one side of the helical mixing element. Mixing effect happens in axial direction at the interfaces between them. The helical mixing element is represented by two cell columns. Some portion of the component A goes to the motion zone of component B and vice versa. Accordingly, kinetics of material motion through the mixing zone is defined by the following matrix equation

$$\mathbf{S}^{i+1} = \mathbf{P}(\mathbf{S}^i + \mathbf{S}_f^i) \tag{III-15}$$

where **S** is a state column vector for the mixing zone, S_f is a column vector of material feed into the mixing zone. The matrix of transition probabilities **P** has the following form according to the model scheme shown in figure III-34:

$$P = \begin{bmatrix} P_{11} & P_{12} & Z & Z & Z & Z \\ P_{21} & P_{22} & P_{23} & Z & Z & Z \\ Z & P_{32} & P_{33} & P_{34} & Z & Z \\ Z & Z & P_{43} & P_{44} & P_{45} & Z \\ Z & Z & Z & P_{54} & P_{55} & P_{56} \\ Z & Z & Z & Z & P_{65} & P_{66} \end{bmatrix}$$
(III-16)

where Z is a zero matrix of size P_{ij} . Matrices P_{ii} are to be built for material motion inside the ith column. Matrices P_{ij} , being upper the main diagonal, are responsible for material motion from the right columns to the left ones, i.e., from the jth columns to the (j-1)th. Matrices P_{ji} staying lower the main diagonal are responsible for material motion from the left columns to the right ones, i.e., from the jth columns to the (j+1)th. Consequently, the matrix of transition probabilities for 6 vertical helical mixing sets and P_{ij} size (4×4) takes form (III-14), where $p_d = (1 - p_c - p_s) / 2$ is for all matrices except P_{11} and P_{66} ; $p_d = (1 - p_s) / 2$ is for the matrices P_{11} and P_{66} ; p_s is a transition probability of components to remain in a cell; p_c is a probability of material to transit in the horizontal direction. Thus, the model has two parameters to identify in the simplest case.

CHAPTER III: ONE AND TWO DIMENSIONAL MODELS FOR STATIC MIXERS



12 columns in the model proposed (6 columns of elements)

Figure III-34: The scheme of the mixing zone (a - arrangement of helical mixing elements, b - a cell model of the mixing zone).

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For all matrices except P ₁₁ and P ₆₆	
$p_d = (1 - p_c - p_s)/2$	

For matrices $~\textbf{P_{11}}$ and $\textbf{P_{66}}$ $p_d{=}(1{\text -}p_s)/2$

n	Δ	0	0	0	Δ	0	0	0	0	0	0	0	Δ	0	0	0	0	0	Ο	0	0	Ο	0
Ps	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
p _d	$\mathbf{p}_{\mathbf{s}}$	0	0	p _d	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
0	p_d	pc	0	0	p_d	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
0	0	1-p _c	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	p _s	0	0	0	p _c	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
p _d	0	0	0	p _d	p _s	0	0	0	p _c	0	0	0	0	0	0	0	0	0	0	0	0	0	0
0	p_d	0	0	0	p _d	0	0	0	0	p _c	0	0	0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	1-p _c	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	n	0	0	0	n	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	Pc	U	0	0	Ps	U	0	0	U	0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	p _c	0	0	P _d	p _s	0	0	P _d	0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	pc	0	0	p _d	0	0	0	p _d	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	1-p _c	1	0	0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0	0	p _s	0	0	0	p _c	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	p _d	0	0	0	p_d	p _s	0	0	0	p _c	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	p_d	0	0	0	p_d	0	0	0	0	p _c	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0	0	0	0	1-p _c	1	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0	0	pc	0	0	0	p _s	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0	0	0	pc	0	0	p _d	p _s	0	0	p _d	0	0	0
0	0	0	0	0	0	0	0	0	0	0	0	0	0	p _c	0	0	p _d	0	0	0	p _d	0	0
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1-p _c	1	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	p _s	0	0	0
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	p _d	0	0	0	p _d	p _s	0	0
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	p _d	0	0	0	p _d	p _c	0
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1-p _c	1

P=

93

(III-17)

It is necessary to note that the matrix **P** has to be normalized so that the sum of probabilities within each column is to be equal to 1, i.e.

$$\sum_{i=1}^{6} \mathbf{P}_{ij} = 1, \ j = 1,2,3,4$$
(III-18)

The last issue included in the model is the procedure of recollection of many material portions, having come from the mixing zone, to the constant sample volumes. This procedure of recollecting microstates into macrostates has been given in clause 1 of chapter III.

2.2 Evolution of the mixture state and its numerical characteristics

2.2.1 Some words about the model and its parameters

The key operator of the mixture evolution is the matrix of transition probabilities. In common case, there are 4 model parameters $p_{sA} p_{sB} p_{cA} p_{cB}$ that depend on the physical properties of components. The size of the matrix may be chosen according to the number of mixing elements installed into the mixing zone. Let us consider the size of the matrix to be 9×12. It means that there are 6 columns and each column can contain up to 8 mixing elements. In general case, this number can be different and it can be chosen according to the design of the axial-crosswise (alternately revolving) static mixer. The state vector defines the concentration distribution of one of the components.

If the matrices P_A , P_B are the same for both components, their distribution will asymptotically tend to be homogeneous, and thus the problem will be to find out the number of revolutions of the mixer to guarantee the required mixture quality.

2.2.2 Study of initial distribution influence on mixture quality

Initial distribution of components influence mixing and mixing time will be different for each initial distribution. In order to investigate this problem, let us consider the case in which transition probabilities are the same for both components A and B. Their initial distribution in the upper container before mixing is shown in figure III-35a for $p_s=0.1$, $p_c=0.3$. This diagram is a concentration distribution in cells in two directions.



column number of cells



Figure III-35 : Process characteristics for $p_s=0.1$, $p_c=0.3$ (a – initial distribution of material in the upper loading container; b – distribution of material after one passage through the mixing zone; c – asymptotical distribution).

The distribution of components in axial and crosswise directions in the lower container after 1 passage is shown in figure III-35b. This distribution is a diagram of concentration distribution in cells in two directions. The surface is horizontal and all sections are filled to the full because the probabilities for the components A and B are equal. Figure III-35c gives almost homogeneous distribution after 10 mixer revolutions.

We will also use a criterion of variance to estimate mixture quality. The formulas for variance calculation are given below.

$$\sigma^{2} = \frac{1}{m \cdot n} \sum_{i=1}^{c} \sum_{j=1}^{m} (c_{i,j} - \langle c \rangle)^{2}$$
(III-19)

$$\sigma_{x}^{2} = \frac{1}{m} \sum_{j=1}^{m} \left(\frac{\sum_{i,j}^{n} c_{i,j}}{n} - \langle c \rangle \right)^{2}$$
(III-20)

$$\sigma_{z}^{2} = \frac{1}{n} \sum_{i=1}^{n} \left(\frac{\sum_{j=1}^{m} c_{i,j}}{m} - \langle c \rangle \right)^{2}$$
(III-21)

where <c> is an average concentration, calculated in the whole volume, <c_j> is an average concentration in the j-th column, <c_i> is an average concentration in the i–th row, m – is the number of columns, n – is the number of rows, σ_x^2 is variance along the horizontal axis x, σ_z^2 is variance along the vertical axis z.

Figure III-36 shows evolution of variance for $p_s=0.1$, $p_c=0.3$ with vertical and horizontal initial distribution of components. Both curves monotonously decrease with the increase of mixer rotations what is expected because probabilities of the compo-



Figure III-36 : Variance evolution after a number of passages for $p_s=0.1$, $p_c=0.3$ (a – vertical initial distribution of components; b – horizontal initial distribution of components).

nents A and B are equal. If the components are placed horizontally (see figure III-36b) the variance decreases more rapidly than in the case of vertical initial distribution of the components (see figure III-36a). After two mixer rotations, we can see that the variance value is $\sigma_A^2 = 0.075$ in case of vertical initial distribution and $\sigma_A^2 = 0.06$ in case of horizontal for the transition probabilities considered.

It is also necessary to make a brief sensitivity analysis of the model parameters. This model has 2 two (p_c and p_s) for each component. Thus if the components have equal physical properties, there are 2 parameters in the model. Let us change them and find the necessary transition number for reaching CV=6% (figure III-37). It can be seen that the variation of the parameter p_s around 0, small values causes significant changes in the process whereas its variation near 1 causes small changes in the process. The same conclusion can be made about p_c . The curves were calculated for the given initial distribution of components, i.e., when the component A is placed at the top and the component B – at the bottom. If the initial distribution is different the curves should differ as well.



Figure III-37: The number of transitions versus different probability values for reaching CV=6% (the components A is initially placed at the top).

Figure III-38 shows how the number of transitions changes if to vary the parameters p_c and p_s for the initial distribution when the component A is placed at the left part of the loading container and the component B is in its right part. Here, the transition number is much less dependent on the change of probability p_s , especially when p_c has higher values. When the value of p_c is higher than 0.2, the curves are almost horizontal, what says that the probability p_s does not affect mixing. We can also observe that the curves almost coincide for $p_c>0.3$, what says that the process becomes less dependent on the value of p_c . If we compare figures III-37 and III-38 we can conclude that the probability p_s influences mixing at a higher extent (figure III-37) whereas p_c influences more in case of figure III-38.

If to have components premixed, less number of rotations is expected to be necessary for having the same value of variance. Figure III-39 gives some view on the evolution of variance with the premixed components. Better mixture quality with less mixing time can be achieved with the



Figure III-38: The number of transitions versus different probability values for reaching CV=6% (the components A is placed on the component B).

initial distribution in figure III-39a than III-39b. The initial scale of segregation is however different in each case so that it is not appropriate to compare the graphs with graphs on figure III-36 precisely.

Figure III-40 shows how total variance changes with different number of mixer revolutions for different initial distribution of the components. The probabilities are p_{sA} = 0.25, p_{sB} = 0.2, p_{cA} = p_{cB} = 0.1 so that a small difference exists in the axial or crosswise mobility of particles. It can be seen that better mixture quality with the less mixing time is reached for the 3-rd initial distribution, i.e. when the component, having higher downward probability, is placed at the top of the loading container. Hence, the model allows estimating how initial distribution influences mixture quality, what is of great practical importance. Therefore, it may be possible to answer the question what initial distribution will give the best mixture quality with the least mixing time after the probabilities are determined.

2.2.3. Study of probability influence on mixture quality

Most of the time, it is necessary to mix components with different physical properties. It means that each component has its own probabilities p_{sA} , p_{sB} , p_{cA} and p_{cB} . Therefore it brings an interest to examine how process parameters change with



Figure III-39 : Variance evolution with the number of passages for $p_s=0.1$, $p_c=0.3$ (a – component A is in the middle; b – component A is in the left lower and right upper corners).



Figure III-40: Variance versus the number of passages for p_{sA} = 0.25, p_{sB} = 0.2, p_{cA} = p_{cB} = 0.1 and different initial distribution of the material components.

different probabilities. In order to examine this matter, let us take a vertical initial distribution of material (figure III-41a). Assuming that the transition probabilities are $p_{sa}=0.1$, $p_{ca}=0.5$, $p_{sb}=0.1$, $p_{cb}=0.3$, i.e., we can say that the component A transits faster in the horizontal direction and more slowly in the vertical direction. The distribution of the components after one and eight passages is given in figures III-41b and

III-41c respectively. The diagrams show that free surface is actually irregular and its inclination results from accumulation of material in that part of the mixer where the component vector with higher probability has been directed to. Evolution of variance corresponding to these probabilities and initial distribution is given in figure III-42a. Variance decreases more slowly than in the previous case when the components A and B had equal transition probabilities. If to increase the probability value of the component A to stay in a cell up to $p_{sa}=0.3$ we will get a variance curve shown in fig-III-42b. In this case the component A transits more slowly in the vertical and ure faster in the crosswise direction than the component B. The variance has much higher values, what says about segregation impact. In addition, the character of variance evolution is oscillating.

Figure III-42c is plotted for equal probabilities $p_{sa} = p_{sb} = 0.1$ but the difference between the probabilities to transit in the crosswise direction is lower than for the



Figure III-41: Process characteristics for $p_{sa}=0.1$, $p_{ca}=0.5$, $p_{sb}=0.1$, $p_{cb}=0.3$ (a – initial distribution of material in the upper loading container; b - distribution of material after one passage through the mixing zone; c – distribution of material after eight passages through the mixing zone).

column number of cells

-8

column number of cells



Figure III-42 : Variance evolution for different transition probabilities (a-c – vertical initial distribution, d-f – horizontal initial distribution).

case in figure III-42a, i.e. $p_{ca} = 0.4$, $p_{cb} = 0.3$. In this case, variance is decreasing faster than that in figure III-42a, and the less the difference in these probabilities is, the faster variance will decrease.

Influence of transition probabilities, when the components are placed horizontally, for $p_{sa}=0.1$, $p_{ca}=0.5$, $p_{sb}=0.1$, $p_{cb}=0.3$ is represented in figure III-42d. The variance curve is oscillating and it has the lowest value $\sigma^2=0.025$ after two passages whereas variance of figure III-42a has the value $\sigma^2=0.08$.

Figure III-42e shows the evolution of variance for $p_{sa}=0.3$, $p_{ca}=0.5$, $p_{sb}=0.1$, $p_{cb}=0.3$. After two passages, the variance has a lower value than for the case of figure III-42b. The same conclusion can be made about the variance evolution shown in figure III-42f for the following values of transition probabilities: $p_{sa}=0.1$, $p_{ca}=0.4$, $p_{sb}=0.1$, $p_{cb}=0.3$.

Thus, a difficult situation can appear that little difference in probability and/or initial distribution may result in a situation when high quality mixture, attained in a short time turns to the constantly segregated state. We can also remark that it is not clear what kind of mechanism is predominant in certain cases. The model captures very different behaviour aspects that are very sensitive to initial conditions.

<u>3 CONCLUSIONS ON CHAPTER III</u>

- One dimensional mathematical model of particulate solid mixing in alternately revolving static mixer and non-revolving static mixer has been developed on the basis of Markov chain theory. The model is able to capture various aspects of the mixing zone design, material feed and specific properties of the components.
- 2. Simulation of mixture quality evolution in alternately revolving static mixers and non-revolving static mixers has been carried out. Segregating and non segregating mixtures were used.
- 3. It has been shown that there is a rational method of material feed and optimal number of material passages through the mixer that provide maximal mixture quality of non-segregating components.

- 4. It has been shown that application of alternately revolving static mixers gives better quality than those of non-revolving type while mixing segregating components.
- 5. An outlook on the mixing time was proposed.
- 6. A two-dimensional model of particulate solid mixing has been developed on the basis of Markov chain theory. The prototype has been the SysMix mixer where mixing occurs both in vertical and horizontal directions. The model takes into account particularities of material loading and different physical properties of the components.
- 7. Numerical experiments have been made. They show how mixture quality changes in alternately revolving static mixers of SysMix type, if the components are not segregating.
- 8. It was shown that while mixing components that tend to segregate, there is a rational way of loading components, and optimal number of revolutions giving the maximal mixture quality. This all can be found by using the model.
- 9. The model proposed allows calculating distribution of components in two directions and estimating mixture quality accurately. Although the model does not take into consideration segregation in the crosswise direction, it can be applied to other apparatus where it is necessary to estimate mixture quality in two directions.

CHAPTER IV:

EXPERIMENTAL STUDY OF STATIC MIXERS

The objective of this chapter is to test the models proposed with some experiments on the equipment. We will develop the algorithm of model parameter identification for each model, and make some experiments with particles of different sizes, densities and shapes.

1 LABORATORY ALTERNATELY REVOLVING STATIC MIXER

1.1 Identification of the model parameters

Identification of the model parameters requires clarification of the following fact. The model parameters are to be found for every new particulate system. In order to perform this task, some simple experiments have to be made with the mixer itself. If one of the components is substituted by another one or another mixer of the same operational principle is used, then it is necessary to make these experiments again and identify the model parameters. Accordingly, the model parameters are connected with the physical equipment and the components to be mixed. There are three parameters to identify in the model: p_A , p_B and α . Briefly, they are determined from two independent experiments: one for p_A , p_B and the other one for α . Here is an algorithm for identification (figure IV-1):

- A particulate system for mixing is taken. The components are put into the loading container so that their initial distribution in the container is known (for instance, top half of the loading container is filled with the component A and the bottom part of the loading container is filled with the component B).
- 2. All mixing elements are removed of the mixing zone and an experiment of free fall is carried out for one material passage. The mass of each component is

measured in every section of the unloading container. Concentration of one component is calculated and the data are put in the table.

- 3. A certain number of mixing sets (e.g. 2) is installed into the mixing zone and the experiments are made according to articles 1, 2.
- 4. The initial state vector is placed into the model and a numerical experiment is



Figure IV-1: Algorithm of model parameter identification.

made. Two–parameter optimization is carried out according to the condition

 $\sum (c_{th} - c_{exper})^2 \Rightarrow min \text{ and}$ transition probabilities p_A and p_B are to be found. At the same time all the delay coefficients $\alpha=1$.

- 5. New values of the transition probabilities p_A and p_B are to be found.
- The delay coefficient α is put in columns corresponding to the number of mixing sets used while performing article 4. The value of the coefficient is taken arbitrary (from 0 up to 1).
- Numerical experiment and one-parameter optimization are performed for the condition

 $\sum (c_{th} - c_{exper})^2 \Rightarrow \min$ and value of the delay coefficient α is to be found.

 After article 7 has been completed successfully, the model is ready for its usage.
1.2 Description of the experimental work

Let us consider the experimental work with the laboratory alternately revolving static mixer ("Lab-made mixer") and the particulate system sugar-couscous. First, the components were put into the loading container (as it is illustrated in figure IV-2), with sugar being on the top and couscous at the bottom. The number of mixing elements required was installed into the mixing zone. Afterwards, the insertion in the upper loading container was quickly removed and the material started falling down through the mixing zone to the unloading container. To measure the mass of each component in every section, insertions were put in (see figure IV-3). Then the lowest insertion was removed and the components were separated with a sieve of 800 microns in diameter for sugar and couscous (It is the sieve size when mixture can be completely separated) and the mass of each component was weighed. The same procedure was done for each of 5 sections. Thus, the mass distribution in the unloading container after one material passage could be estimated. The components were then loaded



Figure IV-2: Distributions of components in containers (a - initial distribution in the upper container, b – distribution in the lower container after one passage).



Figure IV-3: The scheme of the loading container of the "Lab-made" mixer.

as it is depicted in figure IV-2 and a necessary number of mixer revolutions was done. Experiments were made for these components with 1,2,3,4,5,6,9,12,15,19 and 20 mixer revolutions. Every experiment was repeated three times. This procedure was repeated with 5, 10, 20, 40 and 50 mixing elements in the mixing zone. Since the influence of one mixing element on the process was inessential, 5 mixing elements were combined so that they formed a set. Thus, 10 sets of mixing elements could be installed into the mixing zone.

An experiment of free flow was also made when couscous was initially put at the top and sugar was at the bottom. The result of this experiment was the same as if sugar was placed at the top.

Afterwards, the same experiments were made for semolina-millet with the initial distribution shown in figure IV-5.

1.3 Experimental and calculated results

The data shown in figures IV-4, IV-5 are related to the influence of the number of mixing elements and the initial state on the variance evolution of the component A at the bottom. First, mixing was examined in the empty mixing zone without any mixing elements (see the upper left graph). It can be seen that changes in initial distribution of the components has almost no influence on the process kinetics, i.e., the matrix is state independent, and the process is linear. The transition probabilities are the same for both components and the asymptotic distribution is close to the homogeneous. The best fit was obtained for $p_A=p_B=0.91$. The corresponding matrix of transition probabilities was presented as 11x11 matrix: 10 columns for the possible mixing sections and 1 for the accumulating state. So the matrices of transition probabilities are as follows:

$$\mathbf{M}_{A} = \begin{bmatrix} 1 - \alpha_{1} \cdot \mathbf{p}_{A} & 0 & \dots & 0 & 0 \\ \alpha_{1} \cdot \mathbf{p}_{A} & 1 - \alpha_{2} \cdot \mathbf{p}_{A} & \dots & 0 & 0 \\ 0 & \alpha_{2} \cdot \mathbf{p}_{A} & \dots & 0 & 0 \\ \dots & \dots & \dots & 1 - \alpha_{10} \cdot \mathbf{p}_{A} & 0 \\ 0 & 0 & 0 & \alpha_{10} \cdot \mathbf{p}_{A} & 1 \end{bmatrix},$$
(IV-1)



Figure IV-4: Variance for sugar at the bottom versus the number of passages for sugar and couscous with different initial state vectors and different number of mixing elements (p_A =0.91, p_B =0.91, α =0.89).



Figure IV-5: Variance for millet at the bottom versus the number of passages for millet and semolina with different initial state vectors and different number of mixing elements ($p_B=0.94$, $p_A=0.953^*p_B=0.896$, $\alpha=0.89$).

$$\mathbf{M}_{B} = \begin{bmatrix} 1 - \alpha_{1} \cdot \mathbf{p}_{B} & 0 & \dots & 0 & 0 \\ \alpha_{1} \cdot \mathbf{p}_{B} & 1 - \alpha_{2} \cdot \mathbf{p}_{B} & \dots & 0 & 0 \\ 0 & \alpha_{2} \cdot \mathbf{p}_{B} & \dots & 0 & 0 \\ \dots & \dots & \dots & 1 - \alpha_{10} \cdot \mathbf{p}_{B} & 0 \\ 0 & 0 & 0 & \alpha_{10} \cdot \mathbf{p}_{B} & 1 \end{bmatrix},$$
(IV-2)

The data in figure IV-5, IV-6 are related to components with different physical properties (semolina and millet were used). Variance curve is oscillating and it decreases faster if the number of mixing elements is higher. As far as the asymptotic distributions are not even, the matrices for the components are different. The best fit was obtained with $p_B=0.94$, $p_A=0.896$, and, what may be very important, at the same value of the delay coefficient $\alpha=0.89$. This means that the mechanism of the delay could be identical for all the sections in the mixing zone. The revolution of the initial state vector has either no influence on the asymptotic distribution what means that the process stays linear. It is necessary to remember that when p is approaching 1, parameters of the process become very sensitive to its value.

The calculated and experimental results for the case of 4 sets of screws are illustrated in figure IV-6. The model is still able to capture the experimental kinetics.





The CV criteria was calculated and presented on the graphs. The necessary number of mixer rotations to reach CV=6% is indicated on the graphs. If the components have close physical properties, then the more mixing elements is in the mixing zone, the faster it is possible to achieve CV=6% (see figure IV-7).



Figure IV-7: The number of transitions versus the number of sections in the mixing zone for reaching CV=6%.

2 SULZER STATIC MIXER

2.1. Installation scheme. Particularities of mixer feeding

Let us consider the experimental work done with the Sulzer Chemtech static mixer. Firstly, it is necessary to underline some particularities of this mixer and methods of experimentation. The Sulzer mixer works in continuous regime at the industrial plant. The installation scheme is represented in figure IV-8. The mixer can be installed in the flow line when components are being fed or unloaded continuously. At the same time, material is not accumulated but conveyed to the further processing stage (see figure IV-8a). However the Sulzer mixer can be installed at the final processing stage when the ready mixture is accumulated into a silo or packed (see figure IV-8b). Such mixer type has low mean residence time of the particles. It results in application of the mixer mainly for reaching homogeneity in the crosswise direction. However the experimental results, illustrated in [34,35], show that mixing effect in the axial direction also exists.



Figure IV-8: Installation scheme of the Sulzer mixer in processing (a - in-line system; b - mixer installation at the final processing or packaging stage).

2.2 Description of the experimental work

The model developed in this part takes into consideration mixing effect in the axial direction only. It was assumed that components are evenly distributed in the crosswise direction. The purpose was to show that the developed approach to simulation can be used for modeling axial mixing in other static mixers. Besides, it is not possible sometimes to install other mixer types except static ones at some stages of the flow line production. Experimental research has been made similarly to those made with the "Lab-made" mixer, described in clause 1 of this chapter. Material was loaded and unloaded with the usage of loading and unloading containers. Mixer loading is schematically shown in figure IV-9. Although the Sulzer mixer was considered as a batch one, it is not difficult to change the loading type from the batch to continuous. To perform this task, the continuous feeding is to be organized as it is depicted in figure IV-9b.

First, couscous and colored couscous were placed into the loading container as illustrated in figure IV-10. Thus, colored couscous was in the third section beginning from the bottom. A certain number of mixing elements was installed inside the mixing zone (6 is maximally possible).

Mass distribution along the unloading container after one passage could then be obtained. The model described above for such mixer type is represented by the matrix of transition probabilities with the size of 7x7; 6 columns corresponding to 6 mixing elements and 1 column for the accumulating state. The matrices of transition probabilities for each component are:

$$\mathbf{M}_{A} = \begin{bmatrix} 1 - \alpha_{1A} \cdot \mathbf{p}_{A} & 0 & \dots & 0 & 0 \\ \alpha_{1A} \cdot \mathbf{p}_{A} & 1 - \alpha_{2A} \cdot \mathbf{p}_{A} & \dots & 0 & 0 \\ 0 & \alpha_{2A} \cdot \mathbf{p}_{A} & \dots & 0 & 0 \\ \dots & \dots & \dots & 1 - \alpha_{6A} \cdot \mathbf{p}_{A} & 0 \\ 0 & 0 & 0 & \alpha_{6A} \cdot \mathbf{p}_{A} & 1 \end{bmatrix},$$
(IV-3)



Figure IV-9: Initial distribution of components in the loading container of the Sulzer mixer. a – loading container use; b – continuous feeder use.



Figure IV-10: Initial distribution of the components.

$$\mathbf{M}_{B} = \begin{bmatrix} 1 - \alpha_{^{1B}} \cdot \mathbf{p}_{B} & 0 & \dots & 0 & 0 \\ \alpha_{^{1B}} \cdot \mathbf{p}_{B} & 1 - \alpha_{^{2B}} \cdot \mathbf{p}_{B} & \dots & 0 & 0 \\ 0 & \alpha_{^{2B}} \cdot \mathbf{p}_{B} & \dots & 0 & 0 \\ \dots & \dots & \dots & 1 - \alpha_{^{6B}} \cdot \mathbf{p}_{B} & 0 \\ 0 & 0 & 0 & \alpha_{^{6B}} \cdot \mathbf{p}_{B} & 1 \end{bmatrix},$$
(IV-4)

If it is necessary to predict concentration distribution for two or more material passages through the mixing zone, the state vector is not reversed. The state vector is introduced into the mixer inlet in the same order as it was in the lower unloading container after the previous mixing cycle.

2.3 Experimental and calculated results

2.3.1 Non segregating mixtures

The results of experimental work with couscous and colored couscous are represented in figure IV-11. The components only differ by color. Therefore, probabilities for couscous and colored couscous were taken equal. The best fit of calculated and





experimental data is obtained with $p_A = p_B = 0.68$ for 1 and 3 mixing elements and $p_A = p_B = 0.952$ for 6 mixing elements. It is necessary to comment on the different values of the probabilities for the case of 3 and 6 mixing elements. A static mixer can operate in 3 regimes, which were investigated in [34, 35]. Thus, if to install from 1 to 4 mixing elements, the mixer operates in the third regime. If to install 5-6 mixing elements, material flow becomes plugged, as the free space inside the mixer is too small for the same material inflow and the mixer skips to the second operation regime. The transition between the regimes results in higher value of variance $(\sigma_{exp}^2=0.028$ for 6 mixing elements whereas $\sigma_{exp}^2=0.013$ for 3 elements). Obviously, the values of the probabilities are valid only within one regime type. If the mixer changes its operational regime, it is necessary to identify the model parameters once again.

Let us consider another particulate system sugar and couscous. Sugar was also placed into the third section from the bottom. The other sections of the upper container were filled with couscous (see figure IV-12). The best fit of calculated and experimental data is obtained with p_A = 0.65 and p_B = 0.6 for 1 and 3 mixing elements and p_A = p_B = 0.8 for 6 mixing elements (see figure IV-13). The mixer also seems to operate in two regimes, as it was in the previous case.



Figure IV-12: Initial distribution of the components.





Now let us have a look at the experiment when couscous was initially put in the third section and sugar occupied all the rest. The graph corresponding are represented in figure IV-14. It is just enough to set up p_A = 0.6 and p_B = 0.65 in the model and the calculated results approach the experimental ones.

Several experiments were made with the initial distribution of components shown in figure IV-15. The experiments have been made for the following number of material passages through the mixing zone: 1,3,5,9. Every experiment was repeated twice with 1 and 3 mixing elements in the mixing zone. The concentration distribution sugar is represented in figure IV-16. The best coincidence of experimental and theoretical data was obtained with the same values of probability: p_A = 0.65, p_B = 0.6 as for the case of initial distribution in figure IV-15a. The concentration distribution of sugar in couscous is represented in figure IV-17 for the case of initial distribution shown in figure IV-15b. It is worth noting that the results were obtained with the same values of parameters p_A = 0.65, p_B = 0.6, α =0.8.



Figure IV-14: Test experiments with couscous and sugar, 1 passage.



Figure IV-15: Initial distribution of material (a - sugar is at the top, b – sugar is at the bottom).

The graph of variance corresponded to two initial distributions is shown in figure IV-18. The variance curves for 1 and 3 mixing elements when sugar is placed at the top do not differ much. If the sugar is initially placed at the bottom, variance has higher values (e.g. at the point of 2 passages $\sigma_A^2 = 0.06$ when sugar is initially at the bottom and $\sigma_A^2 = 0.04$ when it is on the top). Thus, the model allows predicting mixture quality with different initial distributions of the components to be mixed.



Figure IV-16: Mass of sugar in sections (a - 1 mixing element inside, b - 3 mixing elements inside).



Figure IV-17: Mass of sugar in sections (a -1 static mixer element inside, b - 3 static mixer elements inside).



Figure IV-18 : Variance versus the number of transitions for different initial distributions and different number of mixing elements in the mixing zone.

2.3.2 Segregating mixtures

Some experiments were made with segregating mixtures. Figure IV-19 shows initial distribution of SiC and millet, where SiC was placed in the third section. The experimental and calculated results after one passage are shown in figure IV-20. Better mixture quality is achieved with 3 mixing elements in the mixing zone $(\sigma_A^2 = 0.024 \text{ for 1 element}, \sigma_A^2 = 0.015 \text{ for 3 elements and } \sigma_A^2 = 0.028 \text{ for 6 elements})$. If the number of mixing elements is equal to 6 then we can see that the mixer prepares worse mixture what can be explained by low free space inside the mixer 74% in comparison with empty mixer (see figure II-8). It results in the transition of the mixer from the third operation regime to the second one. Evidently, that in order to describe the new regime, the probabilities have to be restored once again.

Some more experimental results with different components are given in appendix. The results show that the model gives satisfactory prediction of mixture characteristics as variance or concentration for different initial distributions. If the mixer operates within one regime, then the model also gives a good prediction of the characteristics mentioned. The model works with components of different properties and allows taking into account segregation.



Figure IV-19: Initial distribution of the components.





3 SYSMIX ALTERNATELY REVOLVING STATIC MIXER

3.1 Identification of the model parameters

Transition probabilities bind the model with equipment and materials to be mixed. Therefore, to investigate different aspects of the process it is necessary to define the transition probabilities in connection with the real equipment and materials. If



output of materrial

Figure IV-21: Representation of the model as a black box model.

we look at this model as a black box model (see figure IV-21), we may have 4 columns at the upper container and 4 columns at the lower container. Thus we have 4 equations and 4 unknown parameters, what makes it possible to find them. First, it is necessary to make an experiment with equipment and obtain concentration distribution. Secondly, it is necessary to find the probabilities by a nucondition merical method for the $\sum (c_{th} - c_{exper})^2 \Rightarrow \min$. The value of minimum we can specify as 10% ratio of the

value $\frac{\sum_{i=1}^{m} |c_{th,i} - c_{exper,i}|}{\sum_{i=1}^{m} c_{exper,i}}$. Therefore, an algorithm is proposed to identify the model

parameters (see figure IV-22):

- 1. A particulate system for mixing is considered. The components of material are placed into the loading container so that their initial distribution in the container is known (for instance, along the axial axis as it is illustrated in figure IV-25).
- 2. Static mixing grids are installed in the mixer and one material passage through the mixing zone is provided. Afterwards, 3 insertions are placed into the unloading container parallel to the vertical plane so that 4 sections are formed. Then, mass of the components is measured in each section of the unloading container.



- 3. The initial state vector is placed into the model. Afterwards a numerical experiment is made and concentration distribution along 4 sections is calculated for the condition $\sum (c_{th} c_{exper})^2 \Rightarrow \min$.
- After article 3 has been completed successfully, the model is ready for using.

3.2 Description of the experimental work

The SYSMIX alternately revolving static mixer was used by Gyenis and Arva for the experiments [25].

Figure IV-22: Algorithm of model parameter identificer s cation.

It is necessary to examine concentration distribution along two axes, so thay the model also has to give the information about the concentration in axes x and z (see figure IV-23). Quartz sand – sodium chloride, wheat flour – polypropylene granules were used as components by the authors [25]. As far as average diameters and densities are different, the components seem to segregate. Methods of experimentation



Figure IV-23: Axes of mixing.

homogeneity.

with this mixer are described in Chapter II. In addition it can be said that concentration was measured first in vertical and then horizontal directions. As far as sampling destroys the resulted concentration pattern, the experiments corresponding to various mixing times were always started from the very beginning. Variance along the whole volume and variance along the horizontal and vertical directions were the characteristics of the mixture

3.3 Experimental and calculated results

Therefore, there are 4 unknown parameters in the model: the probability of a component to remain in a cell and a probability of a component to transit between mixing elements in the horizontal direction, this is for both components. In order to determine whether the probabilities p_s and p_c are independent from each other, it is necessary to know if the horizontal particle migration influences variance distribution in the vertical direction and vice versa. Let us consider an experiment with a horizontal initial distribution used by Gyenis [25], i.e. it is in the middle of the upper loading container (see figure IV-24). The probabilities are p_{sA} =0.2, p_{sB} =0.3. Now, let us change the probabilities p_{cA} and p_{cB} from 0.05 to 0.5 and see how probability change influences variance in the vertical direction (see figure IV-24). It is clear that transition probabilities of particles in the horizontal direction p_{cA} and p_{cB} significantly influence variance in the horizontal direction for different number of material passages through the mixing zone.

Let us take initial distribution of the components in the center of the upper loading container as it is illustrated in figure IV-25 on the left. The probabilities are taken as $p_{cA} = p_{cB} = 0.1$. Again, let us change the probabilities p_{cA} and p_{cB} from 0.05 to 0.5 (see figure IV-25). As previously, probabilities of particle motion in the vertical direction p_{ca} and p_{cb} significantly influence variance in the horizontal direction.

Let us regard experimental data obtained in [25] and compare them with the results of mathematical simulation. Process characteristics are represented in figure IV-26 for p_{sA} =0.02, p_{sB} =0.3 p_{cA} = p_{cB} = 0.1 (a – initial distribution of the material in the upper loading container; b – variance in the horizontal direction (x axis); c – variance in the vertical direction (z axis); d – total variance along the whole volume). The experimental results were obtained for the components quartz sand – sodium. If the key component is homogeneous along the horizontal axis x, the mixture remains homogeneous in this direction after mixer revolutions. It is the reason why variance in the horizontal direction is equal to zero. If that is so, it can be concluded that there is no segregation along this direction. As far as the probabilities to transit in the crosswise direction in the crosswise direction.



Figure IV-24: Variance in vertical direction (z axis) versus the number of passages for p_{sA} =0.2, p_{sB} =0.3.



k, the number of passages

Figure IV-25: Variance in horizontal direction (x axis) versus the number of passages for $p_{sA} = p_{sB} = 0.1.$



Figure IV-26: Process characteristics for p_{sA} =0.02, p_{sB} =0.3 p_{cA} = p_{cB} = 0.1 (a – initial distribution of the material in the upper loading container; b – variance in the horizontal direction (x axis); c – variance in the vertical direction (z axis); d – total variance along the whole volume).

It results from the model structure assumed earlier. The probabilities of the components are different and the value of CV is 24,2% when the passage rise goes to infinity.

Figure IV-27 shows the process characteristics for the case when the mixing zone is empty. The best coincidence of the theoretical and experimental data was obtained with p_{sA} =0.01, p_{sB} =0.12 p_{cA} = p_{cB} = 0.05. Comparing values of the probabilities we can say that in the last case the downward probabilities are higher than the ones in the horizontal direction. The value of CV is 11,1% when the passage rise goes to infinity, what is lower than in figure IV-26. This difference can be explained that the value of the probabilities p_{sA} and p_{sB} .



Figure IV-27: Process characteristics of the mixer without static mixer elements for p_{sA} =0.01, p_{sB} =0.12 p_{cA} = p_{cB} = 0.05 (a – initial distribution of the material in the upper loading container; b – variance in the horizontal direction (x axis); c – variance in the vertical direction (z axis); d – total variance along the whole volume).

Figure IV-28 gives the experimental and calculated results on concentration distribution in columns. The concentration distribution becomes more homogeneous after 3 passages. This figure is helpful while identifying the model parameters.

Again, the model allows estimating how initial distribution influences mixture quality, what is of great practical importance. In other words, it should be possible to answer the question what initial distribution will give better mixture quality with the least mixing time just by using simulation.



Figure IV-28: Concentration distribution in columns for p_{sA} = 0.2, p_{sB} = 0.3, p_{cA} = p_{cB} = 0.1 (a – after one passage, b – after 3 passages).

4 CONCLUSIONS ON CHAPTER IV

- 1. The procedures of model parameter identification were proposed for the "Labmade", Sulzer and Sysmix mixers.
- 2. Experimental results were proposed for these static mixers.
- 3. It was shown that the models can predict characteristics of mixture quality after any number of mixer revolutions for any initial distribution of the components. As we assumed while identifying the parameters the difference between calculated and experimental results should be not more than 10%.
- 4. The 2D model takes into consideration segregation in the vertical direction.

GENERAL CONCLUSIONS

Today's technology employs more and more complex processes using particulate solids as a mixture. This is relevant to chemical, pharmaceutical, foodstuff, building and other areas of industry. In this respect, a range of components to be mixed and requirements to mixture quality are constantly rising. Investigation of basic material properties and conditions of achieving such quality can be done through experimental methods. However, related cost is high and the reasonable question appears how to cut the cost. In cause of that, the importance of simulation is increasing very fast as far as modeling can reduce the amount of experiments to be done to predict the basic mixture characteristics.

The objective of this work was the improvement of the prediction reliability of theoretical methods by application of Markov chains to simulate mixing process. The aim was an application of the data obtained to development of methods of quality calculation for static mixers.

Three different types of static mixers were considered to develop the models. The mixers of Sulzer type find their application more in contacting materials together rather than in mixing. However, these static mixers are recommended to be installed at the inlet of the continuous drum or blade mixers and other apparatus where several bulk flows are used.

An approach to mathematical modeling has been developed on the basis of Markov chain theory. It allows developing one and two dimensional models of batch static mixers and also other apparatus where macrostates are transformed into microstates. The model works with components of different physical properties.

In particular, a one dimensional model of particulate solid mixing has been developed for static mixers of revolving and consecutive type. The model can predict mixture quality and also optimal number of material passages through the mixer that

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gives maximal mixture quality, even for components having tendency to segregation. The model gives an opportunity to see how initial conditions, like the initial distribution of components or the number of mixing elements in the mixing zone, influences mixing.

As far as one dimensional model only takes into account axial mixing, a twodimensional model of particulate solid mixing has been developed for an alternately revolving static mixer (SysMix). The model allows calculating concentration distribution and variance both in vertical and horizontal directions. By using the model, it is possible to investigate how initial displacement of components influences mixture quality after a number of rotations. It takes into consideration different physical properties of components as well as their tendency to segregation.

Different materials were used to investigate the mixing process in the mixers considered. Algorithm of model parameter identification was suggested. The results of the experiments proved the adequacy of the model developed and allowed to identify parameters of the models. Results of simulation as well as experiments showed that there is an optimal or reasonable number of material passages through the mixer that gives maximal mixture quality with segregating components. It was shown that the processes in these mixers can be described by linear models if the mixer constantly works within one regime.

Some perspectives are arising from this work:

- The sensibility study of the model to the parameters can be done. If the probabilities approach 1 then the process becomes very sensitive to the probability change so that insignificant change in probability values may cause significant change in the results of the process characteristics. This needs a profound study and may lead to technological improvement of mixing element design.
- Experimental study of some more powders can be made. It is also interesting to search for some more combinations of powders that differ by size, by shape or by size and by shape.

- A study of the material surface can be made after feeding the alternately revolving static mixer. It concearns measuring the angle of repose of the bulk in the loading container after its filling. The angle will be different for the components with different physical properties. This will allow having information about initial distribution of components that better corresponds to the reality.
- Experiments of continuous mixing in Sulzer mixer should be also performed. Though this mixer has a low mean residence time, it is possible to study how the mixer reduces the inflow fluctuations and how it mixes in the crosswise direction. The model should be modified to continuous feeding and the results of mathematical modeling should be verified with the experiment.
- A non-linear case with a static mixer of "Sulzer" type may be studied when probabilities become dependent on the feed rate, for example. The mixer can transit from one regime to another one what should affect the values of transition probabilities. In practice, it may happen when the mass flow rate fluctuates to the great extent.
- An influence of mixer rotation speed can be also taken into account in the Sys Mix mixer.

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1 SIEVES AND BALANCE USED FOR EXPERIMENTATION

The photograph of the sieve is shown in figure 1. The electronic balance Sartorius (AG GOTTINGEN LA3200D, 15902097 CULCULUS, Germany Listed scale 261F) was used. Its accuracy is 0.001g. The photograph of the balance is illustrated in figure 2.



Figure 1: The sieve.

Figure 2: The balance Sartorius.

2 Particle size distribution

Particle size distribution was measured by sieving. To separate particles, sieves of different dimensions were used: 200, 400, 500, 630, 800, 1000, 1250, 1600, 2000 and 2500 microns (78312 MAUREPAS.FRANCE AFNOR NFX 22967). The results of measurements are shown in table 1 and figures (1-8).

Table 1: Particle size distribution

millet, totally 60g										
sieve, microns	1250			1600			2000		2500	
mass, g	0			1,394			34,857		60	
passed through the sieve, %	0			2,323			58,095		100	
SIC, totally 100g										
sieve, microns	300			500			630		800	1000
mass, g	0			19,177			94,017		99,743	100
passed through the sieve, %	0			19,177			94,017		99,743	100
aluminum oxide, totally 100 g										
sieve, microns	800				1000			1250	1600	2000
mass, g	0				5,729			5,204	98,652	100
passed through the sieve, %	0				5,729			5,204	98,652	100
sugar, totally 50 g										
sieve, microns	200		31	5	400	50	00	630	800	1000
mass, g	0 0,		0,43	33	0,928	2,2	69 18,397		48,872	50
passed through the sieve, %	0 0		0,86	6	1,856	4,5	538 36,79		97,744	100
couscous, totally 50 g										
sieve, microns	630			800		1000		1250	1800	
mass, g	0				0		0,866		38,186	50
passed through the sieve, %	0				0,000			,732	76,372	100
semolina, totally 50 g										
sieve, microns	80 100			200			315	400	500	
mass, g	0	0 0,181			4,847			l,988	39,482	49,419
passed through the sieve, $\%$	0	0,362			9,694			3,976	78,964	98,838



Figure 6: Particle size distribution of sugar.



Figure 7: Particle size distribution of Couscous.



Figure 8: Particle size distribution of semolina.

3 Additional experimental and calculated results

Let us have a look when millet was put into the third section of the loading container and all others were filled with SiC. The curves are represented in figure 9. It is obvious that as long as we consider the motion of millet, the probabilities should be the opposite to what was in the previous case i.e. $p_A=0.6$ and $p_B=0.7$. The probabilities are equal to $p_A=p_B=0.9$ for the case of 6 mixing elements inside. This is a very important conclusion, that having knowledge about probabilities of the components at once, the distribution of the key component can be calculated along the unloading container. Two sections at the top were initially filled with millet and three bottom sections were field with SiC (see figure 10). After one passage, the distribution becomes as it is shown in figure 11. Flow through the Sulzer mixer must be only in one direction, consequently, the components are to be put in the same order after a passage as they were collected in the unloading container. Results obtained from these



Figure 10: Initial distribution of components.

Figure 11: Distribution of the components after 1 passage.

experiments are shown in figures 12-13. The best fit of theoretical and empirical data was obtained with $p_A=0.7$, $p_B=0.6$. It means that SiC has higher probability to fall. If there is plug flow (in our case this regime corresponds to 5,6 mixing elements), the probability of the components to fall changes to higher values. Probabilities $p_A=p_B=0.9$ return the best coincidence with the experimental data. Now let us have a look how the model predicts mass distribution of the key component if the initial distribution is different. The opposite initial distribution of the components in the loading container is taken i.e., two bottom sections were occupied with millet and the upper sections were filled with SiC. The results are represented in figures 14-17. As it appears from the graphs, the model yields satisfactory mass distribution with different initial distributions and different number of mixing elements. The same probabilities



were used in these tests, i.e., $p_A=0.7$, $p_B=0.6$. for 1,3 mixing elements and $p_A=p_B=0.9$ for 6 mixing elements.

Figure 12: Mass of SiC in sections (1 static mixer element inside).



Figure 13: Mass of SiC in sections (3 static mixer elements inside).



Figure 14: Mass of SiC in sections (6 static mixer elements inside). Millet was initially placed on the top.



Figure 15: Mass of SiC in sections (1 static mixer elements inside). SiC is initially placed on the top.



Figure 16: Mass of SiC in sections (6 static mixer elements inside). Millet was initially place on the top.



Figure 17: Mass of SiC in sections (6 static mixer elements inside). SiC was initially placed on the top.

Another outline of these experiments was carried out with semolina and aluminium oxide. Aluminium oxide has higher density and bigger diameter than semolina. In the first tests aluminium oxide was placed in the third section of the feeder (figure 18)



Figure 18: Initial distribution of the components.

while others were filled with semolina. After one passage, the mass of aluminium oxide is measured in each section of the collector. The empirical results and the results of simulation are shown in figure 19. The matrix of transition probabilities was different for aluminium oxide and semolina what seems reasonable. The best coincidence was observed with $p_A=0.7$ and $p_B=0.6$. The probabilities were taken equal to each other $p_A= 0.96 p_B= 0.9$ for the case of 6 static mixer elements inside. Now let us consider the experiment in which semolina was initially put in the third section and aluminium oxide occupied all the rest. The graph corresponded is represented in figure 20. In order to make calculations with the model it is just enough to change the probabilities. So in this case $p_A=0.9$ and $p_B=0.96$ if there are 1,3 mixing elements inside the mixer and $p_A=0.9 p_B=0.96$ if there are 6 mixing elements. Now let us check how the model works if the initial distribution of the components is different (see figure 21). As soon as aluminium oxide has a higher particle size than semolina, a problem of packing appears. If three sections are occupied with



Figure 19: Test experiments with semolina and aluminium oxide.



Figure 20: Test experiments with aluminium oxide and semolina.

semolina and two sections are occupied with aluminium oxide then after one passage only four sections are filled with material which is not acceptable in our case. To overcome this problem, it was decided to fill two upper sections with mixture of the components. The graphs of simulation and experimental results are shown in figures 22 - 24. The results were obtained with the probabilities $p_A=0.6$, $p_B=0.7$ for the case of 1 and 3 mixing elements. Probabilities $p_A=0.9$ $p_B=0.96$ gave the best fit for the case of 6 elements inside.



Figure 21: Distribution of components (a – initial, b – after 1 passage).



Figure 22: Mass of aluminium oxide in sections (6 mixing elements inside). Aluminum oxide was initially place on the top.



Figure 23: Mass of aluminium oxide in sections (3 static mixer elements inside). Aluminium oxide is initially place on the top.



Figure 24: Mass of aluminium in sections (6 static mixer elements inside). Aluminium oxide was initially place on the top.

The results were obtained with the probabilities $p_A=0.6$, $p_B=0.7$ for the case of 1 and 3 mixing elements. Probabilities $p_A=0.9$ $p_B=0.96$ gave the best fit for the case of 6 elements inside.

Now let us change the initial distribution the opposite way (see figure 25). Thus, in this case the mixture with semolina and aluminium oxide is put in the bottom sections. The results are shown in figures 26 - 28. It is fair to see that the model gives distribution close to experimental one with the same probabilities that has been determined once for all.



Figure 25: Distribution of components (a – initial, b – after 1 passage).



Figure 26: Mass of aluminium oxide in sections (1 static mixer elements inside). Semolina was initially place on the top.



Figure 27: Mass of aluminium oxide in sections (3 static mixer elements inside). Semolina was initially place on the top



Figure 28: Mass of aluminium oxide in sections (6 static mixer elements inside). Semolina was initially place on the top.

4 Programs of models in the computer code Matlab 6.5

4.1 A program of axial flow simulation through a pipe (laboratory static mixer)

The absorber is represented as a stock one. The stock feeder is the turned stock absorber stock feeders and absorber are transformed to the ones of fixed volume

```
S01=[0;0;0;0;0;0;0;0;0;0;0];SS1=[];SS2=[];Sfeeder1=[];Sfeeder2=[];stock1=[]:stock2=[]:
S1=[]:S2=[]: % clarifying variables
S02=[0;0;0;0;0;0;0;0;0;0;0];b1=0;b2=0;A=0;B=0;SSSS1=[];Sprint3=[];nnz1=[];CS=[];
sstock1=[];sstock1=[];Sprint2=[];var=0; variance=[]; % clarifying variables
P1=[];P2=[];g=[];screw=0;a=[1 1 1 1 1 1 1 1 1]; rtd=0;tm=0;
                               % the number of transitions
n=500:
                               % the number of rotations of the pipe
k=10;
for i=1:k;q(i)=i;end
c=10;
                               %
v1=0.3:
                               % velocity of particles to go forward (to fall)
               % calculating the velocity of particles to fall in accordance with the equation
v2=v1%0.8;
Sfeeder1=[0 0 0 0 0 1 1 1 1 1]:
                                   % building the initial feeder. Introducing 0.5 of A
Sfeeder2=[1 1 1 1 1 0 0 0 0 0];
                                   % building the initial feeder. Introducing 0.5 of B
                                   %input('the number of screws= ');
screw=10;
for i=1:round(screw/5)
                                   %coefficients of screws available in each section; if there
% are no screws in a section then a=1:
  a(i)=0.89;
end
initcons=sum(Sfeeder1)/c;
                                  % initial concentration
initvar=0;
for i=1:c
initvar=initvar+(1/c)*(Sfeeder1(i)-initcons)^2;
end
std(Sfeeder1,0)
% the matrix for the first type of particles flowing through the mixer
P1=[1-a(1)*v1 0
                       0
                              0
                                     0
                                            0
                                                   0
                                                          0
                                                                 0
                                                                        0
                                                                                0
                                     0
                                            0
                                                   0
                                                          0
  a(1)*v1
           1-a(2)*v1 0
                              0
                                                                 0
                                                                        0
                                                                                0
  0
          a(2)*v1
                   1-a(3)*v1 0
                                     0
                                            0
                                                   0
                                                          0
                                                                 0
                                                                        0
                                                                                0
  0
                 a(3)*v1
                          1-a(4)*v1 0
                                                   0
                                                          0
                                                                 0
                                                                        0
                                                                                0
          0
                                            0
  0
          0
                         a(4)*v1 1-a(5)*v1 0
                                                   0
                                                          0
                                                                 0
                                                                        0
                                                                                0
                 0
                                a(5)*v1 1-a(6)*v1 0
                                                                                0
  0
          0
                 0
                         0
                                                          0
                                                                 0
                                                                        0
  0
          0
                 0
                         0
                                0
                                       a(6)*v1 1-a(7)*v1 0
                                                                 0
                                                                        0
                                                                                0
  0
          0
                 0
                         0
                                              a(7)*v1 1-a(8)*v1 0
                                                                                0
                                0
                                                                        0
                                       0
  0
          0
                 0
                         0
                                0
                                       0
                                                     a(8)*v1 1-a(9)*v1 0
                                              0
                                                                                0
  0
          0
                 0
                         0
                                0
                                       0
                                              0
                                                     0
                                                            a(9)*v1 1-a(10)*v1 0
                                0
                                       0
  0
          0
                 0
                         0
                                              0
                                                     0
                                                            0
                                                                   a(10)*v1 1];
% the matrix for the second type of particles flowing through the mixer
                                                                        0
                                                                                0
P2=[1-a(1)*v2 0
                       0
                              0
                                     0
                                            0
                                                   0
                                                          0
                                                                 0
  a(1)*v2 1-a(2)*v2 0
                               0
                                     0
                                            0
                                                   0
                                                          0
                                                                 0
                                                                        0
                                                                                0
          a(2)*v2 1-a(3)*v2 0
                                                   0
                                                          0
                                                                                0
                                     0
                                            0
                                                                 0
                                                                        0
  0
  0
                 a(3)*v2
                          1-a(4)*v2 0
                                                   0
                                                          0
                                                                 0
                                                                        0
                                                                                0
          0
                                            0
  0
                         a(4)*v2 1-a(5)*v2 0
                                                   0
                                                          0
                                                                 0
                                                                        0
                                                                                0
          0
                 0
  0
          0
                 0
                         0
                                a(5)*v2 1-a(6)*v2 0
                                                          0
                                                                 0
                                                                        0
                                                                                0
```

```
0
          0
                 0
                         0
                               0
                                      a(6)*v2 1-a(7)*v2 0
                                                                       0
                                                                              0
                                                                0
  0
          0
                 0
                         0
                               0
                                             a(7)*v2 1-a(8)*v2 0
                                                                       0
                                                                              0
                                      0
  0
          0
                 0
                         0
                               0
                                      0
                                             0
                                                    a(8)*v2 1-a(9)*v2 0
                                                                              0
  0
          0
                 0
                         0
                               0
                                      0
                                             0
                                                    0
                                                           a(9)*v2 1-a(10)*v2 0
          0
                 0
                               0
                                      0
  0
                         0
                                             0
                                                    0
                                                           0
                                                                  a(10)*v2 1];
% The process
S1=[00000000000]':
                             % clarifying variables
S2=[0000000000]';
                             % clarifying variables
for h=1:k
                             % an external cycle (responsible for rotations)
  Sfeeder1=flipIr(Sfeeder1); % rotation of the Sfeeder1 vector
  Sfeeder2=fliplr(Sfeeder2); % rotation of the Sfeeder2 vector
                             % a cycle of the process
for i=1:n
  if i<=c SS1=[Sfeeder1(i) 0 0 0 0 0 0 0 0 0 0 0]; % to inject material from the feeder each
                                               % time instant
         SS2=[Sfeeder2(i) 0 0 0 0 0 0 0 0 0 0 0]'; % building the feed state vector
  else SS1=[0000000000]';
     SS2=[0 0 0 0 0 0 0 0 0 0 0 0]';end
                                               % building the feed state vector
  S01=S1+SS1;
                          % building the state vector for a calculation of the process
                          % building the state vector for a calculation of the process
  S02=S2+SS2;
  S1=P1*S01:
                          % the state vector of the component A (the process itself)
  S2=P2*S02:
                          % the state vector of the component B (the process itself)
  stock1(i)=S1(11)-S01(11); % calculation of the component A falling down into the stock
absorber (like RTD)
  stock2(i)=S2(11)-S02(11); % calculation of the component B falling down into the stock
absorber (like RTD)
end
A=0;B=0;sum1=0;Sfeeder1=[];Sfeeder2=[];
                                              % clarifying variables
                                              % setting a counter variable
i=1;
                           % used only to draw the absorber distribution non-normalized
sstock1=stock1;
sstock2=stock2;
n=i-1;
% beginning of the recollection block
bb=10000; A=0;B=0;j=1;Sfeeder1=[];Sfeeder2=[];
for i=1:(n)
                                     % rearranging all the portions to fixed volumes
  for v=1:bb
    if (A+B)<1; A=A+stock1(i)/bb;B=B+stock2(i)/bb;
    else
         Sfeeder1(j)=A; % subtracting that part from i-th cell of the component A to have the
                                                                                 % sum1=1
        Sfeeder2(j)=B;
                         % subtracting that part from i-th cell of the component B to have the
                                                                                 % sum2=1
      i=i+1;
                        % a counter for building of the Sfeeder vector
      A=0:B=0:end
                       % clarifying variables to start summarizing for a new stockfeeder cell
  end
                     % to add the last sum of B when sum1 is not higher than 1
end
Sfeeder1(j)=A;
Sfeeder2(j)=B;
              % end of the recollecting block
```

```
var=0;
for i=1:c
var=var+(1/c)*(Sfeeder1(i)-initcons)*(Sfeeder1(i)-initcons);
                                                               %calculating variance
end
variance(h)=var;
end
 % calculating tmean
tm=0;
for i=1:n
  tm=tm+sum(i*stock1(i));
end
tm
% the end of the calculating block
                                     % drawing graph by using the bar (pie chart diagram)
figure(2)
Sprint=cat(2,Sfeeder1',Sfeeder2');
colormap([0 1 0;1 0 0]);
bar(Sprint,'stacked');
figure(3)
%subplot(1,3,1);
     colormap(repmat([0 1 0; 1 0 0], c, 1)); % drawing initial distribution if there is no mixing by
                                                                              % using one bar
%bar([1 0 1 0 1 1 0 0 0 1;zeros(1,2*c)],'stacked');
axis([0.5 1.5 0 c]);
 v=0;
                              % drawing normalized absorber distribution using one bar
for i=1:c
   for j=1:2
     v=v+1:
     if rem(v,2)==1 Sprint2(v)=Sfeeder1(i);
     else Sprint2(v)=Sfeeder2(i);end
   end
end
subplot(1,1,1);
     bar([Sprint2;zeros(1,2*c)],'stacked');
     axis([0.5 1.5 0 c]);
  CS0=cumsum(sstock1+sstock2);CS=rot90(CS0,2); % drawing non-normalized absorber
                                                                  % distribution using one bar
C20=cumsum(stock2);C2=rot90(sstock2,2);
for i=1:n
% bar(CS(i),'r');hold on;bar(CS(i)-C2(i),'g');axis([.5 1.5 0 c]);hold on;
end
hold off
% game 1
figure(4)
                              %drawing variance
%subplot(2,2,4);
pov=[1 2 3 4 5 6 7 8 9 10];
tm=[31.61 32.90 34.06 35.4];
%q=q.*tm(4);
plot([0 q],[initvar variance],'-r');hold on
```

arid on hold on; %figure(6) %plot([0 3 6 10],tm); %pov=[1 2 3 4 5 6 7 8 9 10]; %tm=[31.61 32.90 34.06 35.4]; %sigma1=[0.12 0.09 0.06 0.04 0.03 0.02 0.017 0.01 0.0005 0.0001]; %sigma2=[0.11 0.06 0.03 0.02 0.01 0.008 0.007 0.005 0.001 0.001]; %sigma3=[0.1 0.042 0.02 0.011 0.008 0.006 0.003 0.001 0.0005 0.0001]; %sigma4=[0.1 0.038 0.018 0.008 0.004 0.001 0.0005 0.0001 0.00001 0.00001]; %game 2 figure(8) dd1=[1 1 2 6 26]; %sigma=0.18 %sigma=0.14 dd2=[1 1 5 18 50]; dd3=[1 3 9 32 80]; %sigma=0.1 dlina=[5 10 20 40 80]; vrrv=[10 10 10 10 10]; subplot(2,2,4);plot(dlina, (dlina./(dd1+vrrv)),'-r*');hold on plot(dlina, (dlina./(dd2+vrrv)),'-b+'); plot(dlina, (dlina./(dd3+vrrv)),'-go'); grid on

4.2 A program of axial flow simulation through a pipe (Sulzer mixer)

The absorber is represented as a stock one. The stock feeder is the turned stock absorber stock feeders and absorber are transformed to the ones of fixed volume

```
S01=[0;0;0;0;0;0;0;0;0;0;0;0];SS1=[];SS2=[];Sfeeder1=[];Sfeeder2=[];stock1=[];stock2=[];S1=[];
S2=[]; % clarifying variables
S02=[0;0;0;0;0;0;0;0;0;0;0;0];b1=0;b2=0;A=0;B=0;SSSS1=[];Sprint3=[];nnz1=[];CS=[];sstock1=[
];sstock1=[];Sprint2=[];var=0; variance=[];
P1=[];P2=[];q=[];screw=0;sumsum=0;ccc=[];Ca=[];variance=[];var=[];initvar=0;
c=5;
                                      % the number of sections
Vcell=6.28*10^(-6);
                                      % the volume of one cell
                                       % g H*m
ge=9.8;
nu=15*10^(-6);
                                       % air density kg/m3
rog=1.19;
                                       % real density kg/m3
ros=1470;
                                       % density of the first (tracer component) kg/m2
roa=1042;
diameter=330*10^(-6);
                                       % average diameter of particles, m
                             % input ('the number of static mixer elements (screws)');
screw=6:
                             % the number of rotations of the pipe
k=12;
v1=0.95;
                             % velocity of first component particles to go forward (to fall)
v2=0.5:
                             % velocity of second component particles to go forward (to fall)
diameter=560*10^(-6);
                             % average diameter of particles, m
Sfeeder1=[1 1 0.5 0 0];
                             % building the initial feeder. Introducing 0.5 of the component A
Sfeeder2=[0 0 0.5 1 1];
                             % building the initial feeder. Introducing 0.5 of the component B
a=[1/v1 1/v1 1/v1 1/v1 1/v1]; % it sets the number of elements not equal to zero
                                    % if there are no screws in a section then a=1/v1:
for i=1:screw
```

```
a(i)=1;
end
velocity1=diameter*(4*ge*ros/(39*sqrt(nu)*rog))^(2/3)
for i=1:k;q(i)=i;end
% calculating initial consentration
initcons=sum(Sfeeder1(1,:))/c;
% calculating initial variance
initvar=0;
for i=1:c
initvar=initvar+(1/c)*(Sfeeder1(i)-initcons)^2;
end
% the matrix for the first type of particles flowing through the mixer
P1=[1-a(1)*v1 0]
                              0
                      0
                                     0
                                            0
                                                   0
                                     0
  a(1)*v1 1-a(2)*v1 0
                              0
                                            0
                                                   0
          a(2)*v1 1-a(3)*v1 0
                                                   0
                                     0
                                            0
  0
  0
                          1-a(4)*v1 0
                 a(3)*v1
                                            0
                                                   0
          0
  0
          0
                 0
                         a(4)*v1 1-a(5)*v1 0
                                                   0
  0
          0
                 0
                         0
                                a(5)*v1 1-a(6)*v1 0
  0
          0
                 0
                         0
                                       a(6)*v1 1];
                                0
a=[1/v2 1/v2 1/v2 1/v2 1/v2 1/v2];
                             %coefficients of screws available in each section; if there are no
       for i=1:screw
                                                              screws % in a section then a=1;
  a(i)=1;
end
% the matrix for the second type of particles flowing through the mixer
P2=[1-a(1)*v2 0
                              0
                                     0
                                            0
                                                   0
                      0
  a(1)*v2 1-a(2)*v2 0
                                     0
                                            0
                                                   0
                              0
  0
          a(2)*v2 1-a(3)*v2 0
                                     0
                                            0
                                                   0
  0
                 a(3)*v2 1-a(4)*v2 0
          0
                                            0
                                                   0
  0
          0
                         a(4)*v2 1-a(5)*v2 0
                                                   0
                 0
  0
          0
                 0
                         0
                                a(5)*v2 1-a(6)*v2 0
                 0
                         0
  0
          0
                                0
                                       a(6)*v2 1];
% The process
S1=[0\ 0\ 0\ 0\ 0\ 0]';
                             % clarifying variables
                             % clarifying variables
S2=[0\ 0\ 0\ 0\ 0\ 0]';
for h=1:k
                             % an external cycle (responsible for rotations)
  i=1;sumsum=1;
  while sumsum>0.001
                             % a cycle of the process
  if i<=c SS1=[Sfeeder1(i) 0 0 0 0 0 0]'; % to inject material from the feeder each time instant
       SS2=[Sfeeder2(i) 0 0 0 0 0 0]; % building the feed state vector
  else SS1=[0 0 0 0 0 0 0]';
      SS2=[0 0 0 0 0 0];end % building feed state vector
                               % building state vector for calculation of the process
  S01=S1+SS1;
                               % building state vector for calculation of the process
  S02=S2+SS2;
                               % the state vector of the component A (the process itself)
  S1=P1*S01;
  S2=P2*S02;
                               % the state vector of the component B (the process itself)
```

```
stock1(i)=S1(7)-S01(7);
                              % calculation of the component A falling down into the stock
absorber (like RTD)
  stock2(i)=S2(7)-S02(7);
                              % calculation of the component B falling down into the stock
absorber (like RTD)
                                                                                       sum-
 sum=S1(1)+S1(2)+S1(3)+S1(4)+S1(5)+S1(6)+S2(1)+S2(2)+S2(3)+S2(4)+S2(5)+S2(6); % a
                                                                 %condition to guit the cycle
  i=i+1:
                                            % a counter of the cycle
end
A=0;B=0;sum1=0;Sfeeder1=[];Sfeeder2=[]; % clarifying variables
                                            % setting a counter variable
i=1:
sstock1=stock1:
                           % used only to draw the absorber distribution non-normalized
sstock2=stock2;
n=i-1:
% beginning of the recollection block
bb=10000; A=0;B=0;j=1;Sfeeder1=[];Sfeeder2=[];
for i=1:(n)
                                      % rearranging all the portions to fixed volumes
  for v=1:bb
    if (A+B)<1; A=A+stock1(i)/bb;B=B+stock2(i)/bb;
    else
         Sfeeder1(j)=A;
                             % subtracting that part from i-th cell of the component A to have
                                                                               the %sum1=1
                             % subtracting that part from i-th cell of the component B to have
          Sfeeder2(j)=B;
                                                                               %the sum2=1
      j=j+1;
                        % a counter for building of the Sfeeder vector
                        % clarifying variables to start summarizing for a new stockfeeder cell
       A=0;B=0;end
  end
                        % to add the last sum of B when sum1 is not higher than 1
end
Sfeeder1(j)=A;
Sfeeder2(j)=B;
% end of the recollecting block
% the block of volume to mass transforming
for i=1:c
   %Ca(i)=1/(1+(rob/roa)*((1/Sfeeder1(i))-1));
   Ca(i)=roa*(10^3)*Vcell*Sfeeder1(i);
  end
     S=Ca:
% the end of the of the block of volume to mass transforming
% variance calculation block
var=0:
for i=1:c
var=var+(1/c)*(Sfeeder1(i)-initcons)^2;
                                          %calculating variance
end
variance(h)=var;
% the end of the variance calculation block
  end
ccc=(size(Sfeeder1));
c = ccc(1,2);
```

```
%figure(1)
                                    % drawing graph by using the bar (pie chart diagram)
%Sprint=cat(2,Sfeeder1',Sfeeder2');
%colormap([0 1 0;1 0 0]);
%bar(Sprint,'stacked');
figure(2)
%subplot(1,3,1);
colormap(repmat([0 1 0;1 0 0],c,1)); % drawing the initial distribution if there is no mixing by
                                                                               %using one bar
%bar([0 1 0 1 1 1 1 0 1 1;zeros(1,2*c)],'stacked');
%axis([0.5 1.5 0 c]);
v=0;
                              % drawing the absorber distribution normalized using one bar
for i=1:c
   for j=1:2
     v=v+1;
     if rem(v,2)==1 Sprint2(v)=Sfeeder1(i);
      else Sprint2(v)=Sfeeder2(i);end
   end
end
subplot(1,2,2);
     bar([Sprint2;zeros(1,2*c)],'stacked');
      axis([0.5 1.5 0 c]);
        CS0=cumsum(sstock1+sstock2);CS=rot90(CS0,2);
                                                                    % drawing non-normalized
                                                      % absorber distribution by using one bar
C20=cumsum(stock2);C2=rot90(sstock2,2);
subplot(1,2,1);
for i=1:n
  bar(CS(i),'r');hold on;bar(CS(i)-C2(i),'g');axis([.5 1.5 0 c]);hold on;
end
hold off
figure(3)
                              %drawing variance
%subplot(2,2,1);
plot(S,'--k');
hold on
axis([1 5 0 7]);
grid on
figure(4)
plot([0 q],[initvar variance],'k');hold on;
```

4.3 A program of mixing simulation in a motionless mixer (Sysmix mixer)

The number of columns should be even and the number of rows should be more than two.

```
P=[];S=[];SS=[];P11=[];P12=[];v1=[];v2=[];v3=[];v4=[];v5=[];v6=[];v7=[];S0=[];
% to clarify variables used in the program
SSS=[];stock=[];Stock2=[];Sstock=[];S5=[];S6=[];SSS1=[];SSS2=[];SS01=[];SS02=[];
matr1=[];matr2=[];Sfeeder1=[];Sfeeder2=[];
```

SS1=[];SS2=[];Sfeeder1=[];Sfeeder2=[];stock1=[];stock2=[];S1=[];S2=[]; % clarifying variables SSSS1=[];Sprint2=[];nnz1=[];CS=[];sstock1=[];sstock1=[];variance=[];variance2=[];variance3= [];q=[];SIZ=[]; % initial conditions r2=0.; % a probability of material to go between screws r2b=0.: ps=0.; % a probability of material to remain in each cell psb=0.; % the number of rotations k=1 % a probability of material to go into an absorber state have to be <=1-r2 to a=1-r2; %meet a demand of normalization % quantity of transitions g=200; % the number of columns m1=12: n1=9; % the number of rows c=10; % the initial number of elements in the feeder with fixed volume S00=zeros(n1,m1); % creating initial S0 n1 by m1 dimension S01=reshape(S00,n1*m1,1); S02=reshape(S00,n1*m1,1); matr1=ones(c,m1); % creating unitary matrix Sfeeder1=[0 0 0 0 0 0 1 1 1 1 1 1 % creating the state feed vector of the component A 00000111111 000000111111 000000111111 00000111111 000000111111 000000111111 00000111111 00000111111 00000111111];Sfeeder2=matr1-Sfeeder1; % creating the state feed vector of the component B % calculating initial variance for i=1:k;q(i)=i;end initvar=0; initcons=sum(sum(Sfeeder1)/(c*m1)); for i=1:c for j=1:m1 initvar=initvar+(1/(c*m1))*(Sfeeder1(i,j)-initcons)^2; end end % end of the calculation block % initial variance vert/hor calculation block aaaa=sum(Sfeeder1')/m1; bbbb=sum(Sfeeder1)/n1 initvar2=(std(aaaa,1))^2; initvar3=(std(bbbb,1))^2; % the end of the variance calculation block

% the program itself r=(1-ps)/2;% calculation of the probability to go forward % a counter of the cycle for i=1:(n1-1) % a vector to be put on the main diagonal of the matrix P11B v1(i)=ps; % a vector to be put on the diagonal below the main one of the matrix P11B v2(i)=r; end % boundary probabilities v1(n1-1)=1-a;v1(n1)=1;v2(n1-1)=a;P11B=diag(v1,0)+diag(v2,-1);% creation the matrix P11B %P11B=[ps 0 0 0 0 0 0 0 0 just to imagine % r ps000000 0 % 0 r ps 0 0 0 0 0 0 % 0 0 r ps 0 0 0 0 0 % 0 0 0 r ps 0 0 0 0 % 0 0 0 0 r ps 0 0 0 0 0 0 0 0 r ps 0 % 0 % 0 0 0 0 0 0 r (1-a) 0 % 0 0 0 0 0 0 0 a 1]: % a counter of the circle for i=1:(n1-1) v3(i)=r; % a vector to be put on the diagonal below the main one of the matrix P11B end % boundary probabilities v3(n1-1)=0; P21B=diag(v3,-1); % creation of the matrix P11B %P21B=[000000000 just to imagine % r 0 0 0 0 0 0 0 0 % 0r000000 % 00r00000 % 000r0000 % 0000r0000 % 00000r000 % 00000r00 % 0000000000;% calculation of the probability to go forward r=(1-r2-ps)/2; for i=1:(n1-1) % a counter of the circle % a vector to be put on the main diagonal of the matrix P11B v4(i)=ps; v5(i)=r; % a vector to be put on the diagonal below the main one of the matrix P11B end v4(n1-1)=1-a-r2; % boundary probabilities v4(n1)=1; v5(n1-1)=a;P11=diag(v4,0)+diag(v5,-1); % creation the matrix P11B %P11=[ps 0 0 0 0 0 0 0 0 just to imagine % r ps000000 0 % 0 r ps 0 0 0 0 0 0 % 0 0 r ps 0 0 0 0 0 % 0 0 0 r ps 0 0 0 0 % 0 0 0 0 r ps 0 0 0

% 0 0 0 0 0 r ps 0 0 % 0 0 0 0 0 0 r (1-a-r2) 0 % 0 0 0 0 0 0 0 a 1]: for i=1:(n1-1) % a counter of the cycle v6(i)=r; % a vector to be put on the diagonal below the main one of the matrix P11B end v6(n1-1)=0; % boundary probabilities P12=diag(v6,-1);% creation the matrix P11B %P12=[0 0 0 0 0 0 0 0 0 0 just to imagine % r00000000 % 0r000000 % 00r00000 % 000r00000 % 0000r0000 % 00000r000 % 000000r00 % 00000000]; for i=1:(n1-1) % a counter of the cycle v7(i)=r2; % a vector to be put on the diagonal below the main one of the matrix P11B end v7(n1)=0: % boundary probabilities P23=diag(v7,0); % creation of the matrix P11B %P23=[r2 0 0 0 0 0 0 0 0 0 just to imagine % 0 r2 0 0 0 0 0 0 0 % 0 0 r2 0 0 0 0 0 0 % 0 0 0 r2 0 0 0 0 0 % 0000r20000 % 0 0 0 0 0 r 2 0 0 0 % 00000r200 % 0000000r20 % 0 0 0 0 0 0 0 0 0]

P22=P11;P33=P11;P44=P11;P55=P11;P66=P11;P77=P11;P88=P11;P99=P11;P1010=P11; P1111=P11; % preparing building the final matrix

P21=P12;P32=P23;P34=P12;P43=P12;P45=P23;P54=P23;P56=P12;P65=P12;P67=P23;P76=P23;P78=P12;P87=P12;

P89=P23;P98=P23;P910=P12;P109=P12;P1011=P23;P1110=P23;P1112B=P21B;P1211=P 12;P1212B=P11B;P1112B=P21B;

Z=zeros(n1); % creation of the zero matrix with shape n1 P1=[P11B P12 Z Z Z Z Z Z Z Z Z Ζ Ζ P21B P22 P23 Z Z Z Z Z Z Ζ Ζ Ζ Z P32 P33 P34 Z Z Z Z Ζ Ζ Ζ Ζ Ζ Ζ Z Z P43 P44 P45 Z Ζ Ζ Ζ Ζ Ζ Ζ ΖZ Ζ P54 P55 P56 Z Ζ Ζ Ζ Ζ Ζ ΖZ Ζ Ζ P65 P66 P67 Z Ζ Ζ Ζ ΖZ Ζ Ζ P76 P77 P78 Z Ζ Ζ Ζ ΖZ Ζ Ζ Ζ Z P87 P88 P89 Z Ζ Ζ ΖZ Ζ Ζ Ζ Z Z P98 P99 P910 Z Ζ

Z Z Z Z Z Z Z Z P109 P1010 P1011 Z Z Z Z Z ZZZZ Z P1110 P1111 P1112B Z Z Z Z Z Z Z Z Z Z P1211 P1212B]; ps=psb;r2=r2b;a=1-r2; r=(1-ps)/2; % calculation of the probability to go forward % a counter of the cycle for i=1:(n1-1) % a vector to be put on the main diagonal of the matrix P11B v1(i)=ps; v2(i)=r; % a vector to be put on the diagonal below the main one of the matrix P11B end v1(n1-1)=1-a; % boundary probabilities v1(n1)=1; v2(n1-1)=a; P11B=diag(v1,0)+diag(v2,-1);% creation of the matrix P11B % a counter of the circle for i=1:(n1-1) % a vector to be put on the diagonal below the main one of the matrix P11B v3(i)=r; end % boundary probabilities v3(n1-1)=0; P21B=diag(v3,-1); % creation of the matrix P11B % calculation of probability to go forward r=(1-r2-ps)/2;for i=1:(n1-1) % a counter of the cycle % a vector to be put on the main diagonal of the matrix P11B v4(i)=ps; % a vector to be put on the diagonal below the main one of the matrix P11B ∨5(i)=r; end v4(n1-1)=1-a-r2; % boundary probabilities v4(n1)=1; v5(n1-1)=a;P11=diag(v4,0)+diag(v5,-1); % creation of the matrix P11B for i=1:(n1-1) % a counter of the cycle v6(i)=r; % a vector to be put on the diagonal below the main one of the matrix P11B end v6(n1-1)=0; % boundary probabilities P12=diag(v6,-1) % creation the matrix P11B % a counter of the cycle for i=1:(n1-1) v7(i)=r2; % a vector to be put on the diagonal below the main one of the matrix P11B end v7(n1)=0; % boundary probabilities P23=diag(v7,0); % creation of the matrix P11B P22=P11;P33=P11;P44=P11;P55=P11;P66=P11;P77=P11;P88=P11;P99=P11;P1010=P11; P1111=P11; %preparing final matrix P21=P12;P32=P23;P34=P12;P43=P12;P45=P23;P54=P23;P56=P12;P65=P12;P67=P23;P7 6=P23;P78=P12;P87=P12; P89=P23;P98=P23;P910=P12;P109=P12;P1011=P23;P1110=P23;P1112B=P21B;P1211=P 12;P1212B=P11B;P1112B=P21B; Z=zeros(n1);% creation of the zero matrix with shape n1

P2=[P11B P12 Z Z Z Z Z Z Z Z Z Ζ Ζ Ζ Ζ Ζ Ζ P21B P22 P23 Z Z Z Ζ Ζ Ζ Ζ Ζ Z P32 P33 P34 Z Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ P43 P44 P45 Z Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ P54 P55 P56 Z Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ P65 P66 P67 Z Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ P76 P77 P78 Z ΖZ Ζ Ζ Ζ Ζ Ζ P87 P88 P89 Z Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ P98 P99 P910 Z Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ P109 P1010 P1011 Z Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ Ζ P1110 P1111 P1112B 7 Ζ Ζ Ζ Ζ Ζ ZZZ Ζ P1211 P1212B]; SIZ=size(Sfeeder1) S1=zeros(n1*m1,1); S2=zeros(n1*m1,1); for h=1:k % an external cycle (responsible for rotations) Sfeeder1=flipud(Sfeeder1); % rotation of the Sfeeder1 vector Sfeeder2=flipud(Sfeeder2): % rotation of the Sfeeder2 vector for i=1:g % a cycle of the process if $i \le SIZ(1,1)$; % transforming one stock array into the state vector to be added on each transition S5=zeros(n1,m1);% other elements of the state vector added are equal to 0 S5(1,:)=Sfeeder1(i,:); % injection of the component A from the feeder into the first row, % other elements of the state vector added are equal to 0 S6=zeros(n1,m1);S6(1,:)=Sfeeder2(i,:); % injection of the component A from the feeder into the first row, SS1=reshape(S5,n1*m1,1); % reshape the matrix representation to the % column one for the component A SS2=reshape(S6,n1*m1,1); % reshape the matrix representation to the % column one for the component B % The end of the transformation block else SS1=zeros(n1*m1,1); % otherwise if the feeder is empty we add zeros each time %instant SS2=zeros(n1*m1,1);end S01=S1; % building state vector for calculation of the process S02=S2: % building state vector for calculation of the process S1=P1*(S1+SS1); % the state vector of the component A (the process itself) S2=P2*(S2+SS2); % the state vector of the component B (the process itself) % collecting the material into stock absorber SS01=reshape(S01,n1,m1); % reshape the matrix S01 to n1 by m1 dimensions for the %component A SS02=reshape(S02,n1,m1); % reshape the matrix S02 to n1 by m1 dimensions for the % component B SSS1=reshape(S1,n1,m1); % reshape the matrix S1 to n1 by m1 dimensions for the %component A SSS2=reshape(S2,n1,m1); % reshape the matrix S2 to n1 by m1 dimensions for the %component B stock1(i,:)=(SSS1(n1,:)-SS01(n1,:)); % calculation of the component A falling down into % the stock absorber (like RTD) stock2(i,:)=(SSS2(n1,:)-SS02(n1,:)); % calculation of the component B falling down into % the stock absorber (like RTD)

```
% the end of the collecting block
  end
  Sfeeder1=[];Sfeeder2=[];
  for y=1:12
    bb=100000; A=0;B=0;j=1;
  for i=1:g
                                     % rearranging all the portions to fixed volumes
   for v=1:bb
        if (A+B)<1; A=A+stock1(i,y)/bb;B=B+stock2(i,y)/bb; % if the sum1 is less than 1 then
                                                         % we summarize the following cells
                                             % if not then the new feeder cell is being formed
             else
        Sfeeder1(j,y)=A; % subtracting that part from i-th cell of the component A to have the
                                                                                  % sum1=1
       Sfeeder2(j,y)=B; % subtracting that part from i-th cell of the component B to have the
                                                                                  % sum2=1
                        % a counter for building of the Sfeeder vector
      i=i+1;
                        % clarifying variables to start summarizing for a new stockfeeder cell
       A=0;B=0;end
  end
end
% clarifying variables to start summarizing for a new stockfeeder cell
                                 % to add the last sum of A when sum1 is not higher than 1
Sfeeder1(i,y)=A;
                                 % to add the last sum of B when sum1 is not higher than 1
Sfeeder2(i, y)=B;
end
SIZ=size(Sfeeder1);
% variance calculation block
var=0;
for i=1:SIZ(1,1);
 for j=1:SIZ(1,2);
  var=var+(1/(c*m1))*(Sfeeder1(i,j)-initcons)^2;
                                                    %calculating variance
end
end
variance(h)=var;
% the end of the variance calculation block
% variance vert/hor calculation block
aaaa=[];bbbb=[];
aaaa=sum(Sfeeder1')/SIZ(1,2);
bbbb=sum(Sfeeder1)/SIZ(1,1);
variance2(h)=(std(aaaa,1))^2;
variance3(h)=(std(bbbb,1))^2;
% the end of the variance calculation block
end
% SySmix experimental data
datahor=[0.33 0.11 0.105 0.08 0.05 0.02];
datavert=([0.25 0.26 0.2 0.1 0.08 0.05].*initvar);
figure(1)
colormap(repmat([0.78 0.78 0.78;0 0 0],SIZ(1,1),1)); % drawing the initial distribution if there
                                                             % is no mixing by using one bar
for y=1:12
```

v=0; % drawing absorber distribution normalized using one bar for i=1:SIZ(1,1) for j=1:2v=v+1; if rem(v,2)==1 Sprint2(y,v)=Sfeeder1(i,y); else Sprint2(y,v)=Sfeeder2(i,y);end end end end bar(Sprint2,'stacked');hold on axis([0.5 12.5 0 SIZ(1,1)]); grid on figure(2) plot([0 q],[initvar2 variance2],'k');hold on %plot(datavert,'or'); hold on; grid on figure(3) plot([0 q],[initvar3 variance3],'k'); hold on; grid on figure(4) plot([0 q],[initvar variance],'-Sk');hold on; axis([0 k 0 0.25]); grid on

ABSTRACT

Mixing of powders is important in many different industries as it adds significant value to the product. The research on this operation in industry goes through simulation of continuous mixing and mixing in general. The objective of this thesis is a development of a unified model representing samples of the particulate flow in different types of static mixers operating with materials that tend to segregate. One and two dimensional models of static mixers based on the theory of Markov chains are developed and the procedure of model parameter identification is proposed. The results of simulation have shown how the value of transition probabilities and initial distribution of the components influence mixture quality. The experimental results have proved the adequacy of the developed models. It has been shown that there is an optimum number of material passages through the mixer that gives maximal mixture quality while mixing components that tend to segregate.

RÉSUME

Le mélange des poudres est une opération importante dans de nombreux domaines industriels. Sa maîtrise à cette échelle passe par la modélisation de l'écoulement et du mélange de manière globale. L'objectif consiste à développer ici un modèle unifié représentant de manière macroscopique le mélange de poudres dans des mélangeurs statiques de conceptions différentes, même en présence de ségrégation. La théorie des chaînes de Markov sert de base pour la modélisation. Des modèles 1D et 2D sont développés, et la procédure d'identification des paramètres est proposée. Des essais de simulation permettent d'identifier l'influence des valeurs des probabilités de transition, de même que la position initiale des produits. Les résultats expérimentaux montrent la pertinence des modèles développés. Ils mettent en évidence l'existence d'un nombre de passage optimal donnant la meilleure qualité de mélange possible lorsque l'on considère des composants ayant une disposition à la ségrégation.