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Principal component analysis for characterising homogeneity in powder mixing using image processing techniques

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Abstract

The many methods which exist to characterise the quality of a powder mixture have been recently reviewed and linked with mixing mechanisms in current literature. In this paper, we try to develop a novel methodology for defining and characterising homogeneity using principal component analysis (PCA) as an alternative to well-known statistical methods, such as auto-correlation functions or variances. We apply this to image analysis for the case of a powder mixture flowing out of a continuous mixing device. An emphasis is placed on the calculation in real-time of the degree of homogeneity of loose materials on the conveyor belt, carrying this mixture. Mass flow disturbances applied to a binary mixture are studied by the proposed methodology, which is found to be sensitive to small structural defaults.

Keywords: Powder mixing; Homogeneity criterion; Image analysis; Principal component analysis

1. Introduction—state of the art

Powder mixing is a widely used unit operation in the pharmaceutical, agro-food, cement or chemical industries. The process is complex as it depends on many factors operating at various scales: single particle properties, bulk particle properties, general mixer design and operation, combination of operating conditions, mixture formulation, etc. The quality of a mixture, which may be the degree of homogeneity of loose material at the outlet of a mixer, is important for end-user properties as perceived by customers and/or for in-process properties used by manufacturers for normative procedures. In addition, mixing process optimisation is a matter of reducing mixing time and saving electrical energy, especially for low added-value products.

The definition of mixture quality first requires the knowledge of the scale at which it has to be defined, that is the scale of scrutiny of a mixture. For example in the case of pharmaceutical tablet the size of scrutiny is probably equal to a tablet size, that is to say the size of the patients dose. However, this may not be so when the patient interferes, such as when he is allowed to sample himself in the package (if the patient take a piece of tablet for example).

The many methodologies that exist to characterise mixture homogeneity and its structure were recently reviewed and linked with mixing mechanisms by Gyenis [1]. A distinction must be made between the techniques that calculate a structural criterion and those which generate an overall criterion, which are well known in industrial practice. The standard deviation of the composition of a certain number of samples, as well as the corresponding variance, coefficient of variation, or mixing indices belong to this latter category. Weinekotter [2] illustrated the limits of such analyses by discussing two continuous processes having the same variance but radically different organisation: (a) 12 samples with compositions alternating on both sides of the mean; (b) six samples of identical composition higher than the mean followed by six samples of identical composition lower than the mean. When compared to each other from a "customer" end-used property, it is obvious that process (b) is inadequate while it may satisfy a pharmaceutical quality criterion.

On the other hand, fractal analysis, phase-space techniques and the study of auto-correlation functions allows taking into

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account the structure of a powder mixture, as the relative positions of the samples are not destroyed by the analysis. Danckwerts [3] was probably the first to suggest the use of auto-correlation functions to describe the scale of segregation of a mixture, that is the maximum size of segregated regions in a mixture · · · or the sample size above which powder homogeneity is only due to random effects. The auto-correlation function R characterises the interdependence of the compositions of any two samples of a series of samples, separated by a distance r (or by a given number of samples). If no correlation exists, then the value of $R(r)$ is equal to zero and the mixture can be considered as homogeneous at this scale. In other words, the scale of segregation becomes equal to the scale of scrutiny. In practice, the first "distance" r_0 for which R equals zero can be read from a plot of R versus r, called a correlogram. This distance is the parameter for characterising the structure of the mixture, as it gives a measure of the maximum length of no correlation between samples. However, the determination of r_0 is not always easy, at least because a correlogram may exhibit asymptotic behaviour, or because it is very sensitive to a small variations in the composition of the samples. Indeed, there is still a need for developing new, meaningful, industry-pertinent methodologies for quantifying the homogeneity of powder mixtures.

From the point of view of the techniques used for obtaining such information, recent emphasis has been placed on on-line methods which avoid the interference of thief sample probes [4] and the related statistical problems. Optical measurement techniques have received wide attention in the scientific community, especially in the past decade, from the pioneering work of Harwood et al. [5], to that of Berntsson et al. [6] (one may also refer to Weinekötter and Reh [7] or Steinmetz et al. [8]). Near Infrared (NIR) spectroscopy is now beginning to be used in industrial R&D for measuring powder blends. However, many of the workers involved in such projects find difficulties in the calibration procedures and time stability of the signals. Also concerning these type of methods, but very much at the research stage, we may cite laser induced fluorescence as a very promising tool for pharmaceutical mixtures [9]. Electrical capacitance tomography (ECT) has been under rapid development since the mid-nineties and has been applied to real cases in general powder technology [10–12], and in powder mixing in particular [13]. While it seems to be a valuable alternative to optical methods, it seems better suited for coherent pipe flows, that is to say with low disturbance of the flow rates because porosity changes have a strong influence on data reliability. In comparison to other measurement techniques, image analysis is non-destructive and is characterised by a great speed, which is very important in on-line systems. In the recent scientific literature, Muerza et al. [14] have proposed such a method. In this work the method is applied to two free-flow powders differing in colour mixed by a continuous static mixing unit and poured for transport by a belt conveyor without disturbance. A CCD camera placed on the conveying line captures images of the moving belt. These images are then treated and analysed using the auto-correlation technique to obtain the characteristic distance r_0. Because this required a critical analysis of the correlograms, with respect to their shape, it was performed off-line from the measurement chain.

From the above, one may understand that a single method cannot cover the whole range of possible powder blends. Also, when considering its applicability to industrial problems, the exactitude of the method comes after other type of considerations, such as the stability of the measurement with time, easy calibration procedures or no calibration required at all, sampling needed or not, compatibility with industrial conditions, real-time data analysis, ... Therefore, working on new on-line methodologies for accurate data treatment in real-time is still an important field of investigation. This paper proposes a novel method based on real-time principal component analysis of powder mixture images captured by a CCD camera with the claim that this methodology of data processing may be adapted to other types of on-line techniques, such as capacitance measurements or NIR spectroscopy.

2. Powder mixture measurement based on image analysis

The method developed in this paper aims to measure the degree of homogeneity of loose materials on a conveyor belt using image processing techniques. For this, one powder (semolina; particle size, 100–250 μm) and a tracer (semolina coloured by iodine adsorption) were used as particulate systems having identical properties except colour. The measurement chain consists of a CCD camera placed over the conveyor belt which transports the mixture of the loose materials, as well as a PC computer. The schematic diagram of this system is presented in Fig. 1.

A time sequence of images is captured by the camera and represents the solid mixture on the belt during conveying. These images are treated with specially designed computer software to calculate the homogeneity ratio of the mixture. Each image represents a two-dimensional sample of analysed mixture. The pinhole camera model used (see Fig. 2) is detailed in Appendix and provides a continuous brightness function corresponding to each frame. Digitizing a frame consists in sampling and quantifying the function, it gives a grey scale image containing information about tracer concentration. Indeed the black tracer concentration is directly linked with the brightness as its reflectance is different from that of the white particles.

In the example shown on Fig. 3, a sample and a quantized binary mixture image is shown. The diameter ϕ of any particle in the mixture is approximately equal to five pixels.

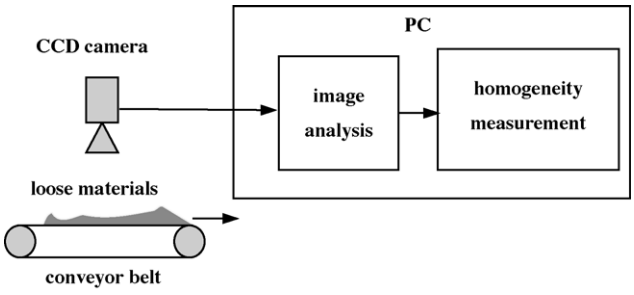

Fig. 1. Schematic diagram of the solid mixture analysing set-up.