Available online at www.sciencedirect.com
SCIENCE DIRECT®
Chemical Engineering and Processing 44 (2005) 303–313
Chemical Engineering and Processing
www.elsevier.com/locate/cep

Assessing the homogeneity of powder mixtures by on-line electrical capacitance

N. Ehrhardt [a], M. Montagne [a], H. Berthiaux [a,*], B. Dalloz-Dubrujeaud [b], C. Gatumel [a]

[a] *Laboratoire de Génie des Procédés des Solides Divisés, UMR CNRS 2392, Ecole des Mines d'Albi-Carmaux, Campus Jarlard, route de Teillet, 81 000 ALBI, France*
[b] *Département de Mécanique Energétique, UMR CNRS 6595, Ecole Polytechnique Universitaire de Marseille, Technopôle de Château-Gombert, 5, rue Enrico Fermi, 13 453 Marseille, France*

Received 27 August 2003; received in revised form 10 March 2004; accepted 19 March 2004
Available online 25 June 2004

Abstract

In this paper, we try to improve our comprehension of one of the most important challenge in the particulate solids industry: the assessment of the homogeneity of powder mixtures. A two-electrode electrical capacitance method is used to measure the permittivity of mixtures while flowing in a 1-D set up. An emphasis is placed on the possibility of following homogeneity evolution through permittivity measurement, without the necessity to calculate the volumetric proportions of each component from an effective medium formulation. The methodology is applied to examine segregation in a funnel, as well as the efficiency of a laboratory drum mixer.

Keywords: Homogeneity; Capacitance; Powder mixing

1. Introduction

With the increased variety of formulated products of the particle form that appears every day on the market and that are issued for a wide range from industry (pharmaceutical, agro-food, cement, plastics, ...), particulate processes in general, and mixing processes in particular, are pushed to a never-ending battle against their own performances. This is due to the fact that the end-used properties of the products that are manufactured nowadays are more and more complex, especially because these must cover more and more functions. Because product engineering had become a major engineering science producing its own tools both at the research and the educational levels, a product ought to become an "intelligent delivery system", a "small process" able to optimise its own action when it is used.

However, it is of common knowledge that mixers, as much other equipment dealing with particles, are all designed and operated from the purest empiricism. Indeed, there exists an important mismatch between the refinement attained at the formulation step and the development of processes able to manufacture the designed and desired product. If we take biodisponibility of a pharmaceutical tablet as an example, the trust that we have in formulation must be contrasted with the following "traditional chemical engineering" interrogation: "How to make sure that the process will deliver pills, all having the same biodisponibility?". In practice, this difficulty in controlling the product has led the companies and some regulatory institutions, such as the FDA, to emphasise on "freezing the process" by avoiding any change. In many process industries, the main keyword concerning powder or granular material mixers is undoubtedly "reproducibility", with the meaning that once an equipment is qualified and validated to perform a certain function, operating conditions are fixed and the cost of any variation in the process parameters may become prohibitive [1]. For example, in the pharmaceutical field, even a slight change in the mixing process would lead to clinical trials for ensuring that the drug's therapeutic effect has not been affected. In addition, current sampling procedures are widely contested [2–4], especially because of the limited number of samples – typically thirty – that can be assumed in the context of production with

* Corresponding author. Fax: +33 5 63 49 30 25.
E-mail address: berthiau@enstimac.fr (H. Berthiaux).

0255-2701/$ – see front matter © 2004 Elsevier B.V. All rights reserved.
doi:10.1016/j.cep.2004.03.016

respect to the overall number of possible samples – about one million.

In other words, the technological improvement of the products does not guarantee the same progress at the manufacturing level at all, mostly because of a lack of instrumentation and control of the processes, more deeply linked to the lack of understanding and reliability of particle characterisation in general. If we except the mineral industry, the industrial applications of particle control systems are still scarce, the only well-known example being particle size distribution measurement through LASER diffraction. However, the situation is not so dramatic if we look at the progress made in the field of particulate instrumentation research in the last decade, with a constant special insight provided by the international conference series "Control of Particulate Processes" [5].

Optical techniques were developed – both in situ and on line – over a broad range of applications and various types of light sources. Main examples are solids concentration measurements in particle fluidisation [6,7], as well as gas–solid flow [8], or sample composition in dry blending operations, this latter beginning from the pioneering work of Harwood et al. [9] up to more recent advances in this scientific field [10–12]. The main interest of these techniques lays in the fact that optical probes are easily available on the market, so that qualitative monitoring of mixers is possible at an industrial scale. However, these probes only provide local information of the mixture (typically 1/40 of a tablet), so that in essence, they still "sample" the powder flow in a way that may be intrusive and not always representative of the overall stream.

Electrical capacitance tomography (ECT), as well as electrical resistance tomography (ERT) for liquid–solid systems, experienced a flourishing development these recent years for a wide variety of applications [13–18]. Their major advantage is that they provide, in a non-intrusive way, a cross-sectional view of a stream that may contain at the same time a liquid, a solid and a gas. In that sense, they are able to give a radial image, which in turn, and through the help of other multiple electrode sensors in the axial direction, can be reconstructed in three dimensions. However, this huge amount of information must be contrasted with the low resolution at which it is given. This has probably limited the application of this technique to powder mixtures that require an analysis at a very fine scale. X-ray, γ-ray, as well as magnetic resonance imaging (MRI), must also be cited in the category of the tomographic techniques. They were mostly used to investigate fluidisation processes, but still seem difficult to employ at the level of a factory [19–21]. LASER-induced fluorescence (LIF) technique must finally be cited, especially because some pharmaceutical ingredients can naturally provide a signal when excited by the adequate beam [22].

In this work, we develop an electrical capacitance sensor that consists of only two electrodes placed around a glass tube. This set up is considered not in the view of imaging the media (such as in classical ECT methods), but with the objective of obtaining a global dielectric information and follow its variation while powder is flowing into the tube, with the aim to measure the composition of binary or ternary mixtures in the axial direction of the powder flow stream. It is waited that this method will provide, through specific iterative algorithms, a quantitative and structural information about a binary mixture flowing out of a mixer (either batch or continuous) in relation with its own processing.

2. Theoretical and conceptual developments

2.1. Powder blend homogeneity: macro and micro considerations

Homogeneity is a key concept for many chemical engineering applications, especially those in which a transformation occur (precipitation, chemical reaction, grinding, ...), because of the coupling between kinetics aspects and contact time of the "reagents" due to the flow conditions. In the field of powder mixing, in most cases, no transformation takes place and the quality of the product is the fact of the particulate flow dynamics itself. Also, the main difference with fluid mixing lays in the fact that a perfect mix cannot be performed at a molecular scale, and is still difficult to assess at the scale of a particle. This is due to a wide variety of particle–particle interactions, resulting in a conflict between mobility of single particles, which is essential to achieve a good mix, and resistance to segregation, which is a fact of the mixture's structure. In that sense, cohesive particulate media (typically below 50 μm) can be viewed as "macrofluids", while free-flowing systems (typically superior to 500 μm) can be viewed as "microfluids". Between these two extreme behaviours – that sometimes may occur in a single product (case of aerable powders) – particle and particle packets trajectories and motion are both influenced by gravity and surface forces.

As felt above, the concept of powder blend homogeneity cannot be dissociated from the scale at which a mixture is observed. For continuous mixers, homogeneity must be defined at the level of the blend flowing out of the vessel, and falling down to a conveyor or any other transportation equipment. In the case of a batch mixer, the mixture structure can be particularly complex to define, but in practice and when the mixer is emptied, the content of the vessel is discharged through a "pipe" to a container or a tableting machine. Hence, in both cases and if we take into account the entire cross-section of the pipe, the mixture flow can be assimilated as a 1-D particle stream.

Thus, let us consider a powder mixture flow as a monodimensional layer divided into N consecutive "elementary" samples. In a first approach, the size of these can be arbitrary fixed, not too small to prevent from statistical errors (inversely proportional to the square root of the particle number) and not too large for ensuring the validity