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Validation of an inline particle probe in a high-shear mixer for particle size determination

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A B S T R A C T

This paper deals with agglomeration processes in a high-shear mixer and the integration of an inline particle probe. The purpose is to determine particle size distribution continuously in real-time. High-shear mixers rotate with a very high mixing tool speed. As a result, not only a mixing effect, but also a granulating effect (under certain conditions) is obtained. This granulating effect is to be studied extensively using an inline particle probe. In the first instance, it is decisive to integrate the probe and find settings to measure and to represent the whole particle size distribution in the mixing vessel. This article will highlight some of the findings obtained from the comprehensive parameter study. Additionally, the technical implementation of the inline particle probe in a high-shear mixer will be described.

1. Introduction

More process analysis technologies (PAT) are not only claimed in the Guidance for Industry [1] of the Food and Drug Administration (FDA), but are rather in the interest of all industries defining products by their physical properties. One goal is the real-time analysis and documentation of manufacturing processes. Results can be used to monitor quality parameters, set target values, and identify and eliminate product defects. The implementation of process analytical technologies bears a great potential for process optimization and understanding as well as enormous cost-saving opportunities. It is important not only to identify fundamental properties, but also to determine the technical behaviour in manufacturing processes.

For determination of process-relevant parameters, four classifications of process analysis are made: offline, atline, inline, and online. Offline and atline analytics are determined by manual or semi-automatic sampling followed by discontinuous evaluation in central laboratories (offline) or in specific analyzers near to the production place (atline). In both cases, the product properties may change during characterization. On the other hand, online and inline procedures permit continuous process control without manual sampling. This means that a continuous correlation between received information and the features of the process or product is possible. For online analysis, in most cases, the sample is measured in a bypass. In the case of inline measurements, the measuring cell is located directly in the product stream so that sampling is eliminated completely.

The particle size distribution of a product is crucial to many of its features and to subsequent processing. Hence, there is an increasing need for its real-time determination. With this information, it is possible to react directly to changes in the product quality. Many investigations to determine the particle size continuously with an inline particle probe have already been carried out in fluidised beds [2,3] and grinding processes [4,5]. Real-time monitoring and control of agglomerations in a high-shear mixer are of importance to science and manufacture. In this way, more information is obtained about the process and about the operating parameters that influence agglomeration. In general, many basic investigations have focused on wet granulation processes [6–10]. More detailed studies consider binder properties [11–15] or growth kinetics [16,17]. Nevertheless, there is still a lack of understanding. This article illustrates the possibility to determine the particle size inline and the interaction between particle stress and different process parameters.

2. Instruments and materials

2.1. High-shear mixer

All experiments were carried out on an Eirich type high-shear mixer R02 manufactured in Hardheim, Germany. The mixer consists of a rotating mixing vessel, a rapidly rotating eccentric mixing tool, and a wall

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scraper. The mixing vessel and the agitator can be operated in the counter- and co-current flow modes. As a result of the mixing vessel's rotation, the test product is transported into the high-shear zone, i.e., towards the mixing tool. The wall scraper is a static tool which prevents deposit build-up on the vessel wall. Products adhering to the vessel wall are detached. Two mixing tools were studied. Fig. 1 shows the star agitator (left) and the pin agitator (right). Due to their different geometries, they also differ in other aspects. The star agitator exerts a radial mixing effect with cutting and impact load. In addition, it fluidises the mixing vessel content. The pin agitator, by contrast, exerts an axial mixing effect with a high shear load on the product which keeps the material more compact. Other features are listed in Table 1 and [18].

2.2. Determination of particle size and particle size distribution

2.2.1. Inline particle probe

The investigated method to determine the particle size is a fibre-optical spatial filtering technique, which is well-studied and still integrated, e.g., in fluidised bed processes [2,3,18–20]. The “Inline Particle Probe 70” has been designed for inline particle size determination in industrial production facilities by Parsum GmbH, Chemnitz, Germany. The system determines simultaneously the time and the velocity of particles passing the probe's sensing volume. It consists of a periodic arrangement of waveguides in the form of a lattice with a characteristic lattice constant. With an optical-fibre spatial-frequency filter anemometer (“ab“differential lattice), particle velocity v_p is determined. For this purpose, moving particles are projected onto the optical lattice. Then, burst detection/frequency analysis of the produced signal is carried out. A supplementary optical channel (“c” monofibre) allows the particle diameter x_p to be determined from the pulse width t_p. The statistical particle diameter x_p is measured directly as the chord length in the direction of the spatial filter axis. The mode and function of the particle probe are described in [2,18–21] in greater detail. The measurement range extends from 50 μm to 6 mm (without additional dispersing unit). Due to the high solid concentration in the process, it is necessary to use an additional dispersing unit, which serves to disperse and dilute particle flow in the sensing volume. Since the entrance window of the dispersing unit has a nozzle with 2.5 mm, the maximum measurable particle size is reduced (from 6 mm) to 2 mm.

2.2.2. Retsch Camsizer®

To assess the agglomerates and validate the inline particle probe, an automatic image evaluation system by Retsch & Technology is applied. The Camsizer® uses the principle of digital image processing. It is qualified for dry analysis of powders and granulates. The measuring range of particles is between 30 μm and 30 mm. The granulate curve can be determined in a non-destructive manner. Digital image processing yields the following characteristics: Median value x_{50}, particle size x_{10}, and particle size x_{90}.

Fig. 1. High-shear mixing tools for the Eirich-type high-shear mixer. Star agitator (left); pin agitator (right).

Table 1
Agitator—technical details.

	Unit	Star agitator	Pin agitator
Diameter of the mixing tool	[mm]	135	125
Impact body	[−]	8	6
Striking surface	[mm^2]	20586	10672
Impact body surface	[mm^2]	22130	3104
Mass	[g]	475	875

2.3. Material

The agglomeration experiments are carried out using cohesive clay and feldspar. These two powders are mixed in a ratio of 3:2 (kaolinitic clay:feldspar). The properties of the test product are listed in Table 2. To trigger the agglomeration process, the binder liquid is added via a dropping funnel directly into the mixing zone. During each mixing experiment, purified water is used as binder liquid.

3. Experimental procedure, test results, and interpretation

3.1. Pilot test

To ensure that the probe works properly and a comparison with other particle size measuring systems is possible, some preliminary tests have to be made. The particle sizes and particle shapes of the small glass marbles ($x = 0.75\ldots1$ mm) and dry quartz sand ($x = 0.15\ldots1$ mm) used are similar to those of the agglomerates examined at a later stage. Most pilot tests are performed in a simple test setup and not in the high-shear mixer itself, because the mechanical stress caused by glass marbles, in particular, is too high. In the pilot test setup, the particles fall freely through a pipeline and directly through the probe measurement volume. As a result, particle flow is very homogeneous and constant. For the glass marbles, the comparison of the inline particle probe, the Laser Diffraction Sensor (Helos by Sympatec) and the Camsizer has revealed a very good match of the cumulative distribution. The results for the dry quartz sand are shown in Fig. 2. Based on the higher mechanical stress of the quartz sand, it was possible to calibrate the particle size directly in the mixer, too. The values obtained at an agitator speed of 500 rpm (IPP-70 dynamic) are shown. The results agree with the experiments performed outside of the mixer (IPP-70 static). The observed differences are due to the different measuring methods. As a result, the Inline Particle Probe and the Camsizer have the chord length of the particle. The diameter of the Laser Diffraction Sensor is equivalent to that of the particles. These differences arise because quartz sand is an irregularly shaped product. It is very important to realize that the results obtained using the inline particle probe are in-between those obtained by the two established methods. Further

Table 2
Test product properties.

	Unit	Kaolinitic clay	Feldspar	Mixture
Colour	[−]	Grey	White	Grey
Particle diameter x_{10}	[μm]	1.09	1.49	2.29
Particle diameter x_{50}	[μm]	4.41	12.26	6.30
Particle diameter x_{90}	[μm]	22.40	41.05	32.16
Specific surface S_V	[m^2/cm^3]	2.27	1.43	1.79
Solid density ρ_S	[g/cm^3]	2.66	2.62	2.64
bulk density ρ_{bulk}	[kg/m^3]			696.5