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A comprehensive shape analysis pipeline for stereoscopic measurements of particulate populations in suspension

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ABSTRACT

A state-of-the-art, compact optomechanical setup coupled with an image analysis routine to measure multi-dimensional particle size and shape distributions (*n*D PSSDs) for crystallization processes is presented. A novel image processing pipeline to process the raw images from the cameras is presented. The pipeline consists of a stereoscopic camera calibration model, adaptive background subtraction, particle contour matching, and 3D reconstruction of the segmented crystals. The reconstructed crystals are subjected to a supervised shape classification strategy, which categorizes each detected crystal into spheres, needles, quasi-equant particles, platelets and non-convex particles. Additionally, a high-speed image capture mode, capable of monitoring processes with fast kinetics, is presented. The device discussed in this work is subjected to an experimental campaign, to validate size measurements, characterize steady state, and confirm repeatability of measurements to affirm and assess the non-invasive nature of the setup on the measurement. An experiment aimed at evaluating the enhancement in the proposed image analysis pipeline performance, more specifically the automatic shape classification, is further conducted. Finally, a dissolution process is monitored using a stereoscopic imaging setup for the first time, and the size and shape evolution of the population in a growth and dissolution phase is monitored for about 18 h.

1. Introduction

In crystallization processes, the particle size and shape of the final product is of key importance as it influences the downstream processing operations such as filtration, drying, and tableting. Crystals exhibit different shapes depending on the crystal habit, and an accurate characterization of shape is critical in the design and control of such processes. Often, commercially available crystallization process characterization tools condense shape related information of crystals into a single characteristic length [1]. Hence, a one dimensional particle size distribution is obtained leaving out the shape information of the crystals. However, due to the variety of shapes exhibited by crystals, in order to accurately quantify the population of crystals, a *multidimensional* particle size and shape distribution (PSSD), rather than a PSD, would be preferable.

As we have recently reported [2], commercially available sizing tools that rely on the assumption of a single characteristic length, such as focused beam reflectance measurement (FBRM), laser diffraction (LD), Coulter counter (CC), as well as monoscopic imaging tools, are prone to errors and misleading effects for particles that are non-spherical. For example, FBRM provides a one-dimensional chord length distribution (CLD) which is difficult to interpret; the transformation from the CLD to PSD is an ill-posed problem and can in fact simply not be done without additional tools [3]. Multi-projection imaging systems [4–7], have been proposed as remedy as they are able to tackle shape-related issues rather satisfactorily, thus reducing the ill effects encountered by commercial sizing tools for non-spherical particles.

In an earlier publication, the successful implementation of a dual projection imaging device using a stereoscopic camera setup with a sapphire glass based flow channel was demonstrated [8]. The setup has been used to monitor growth and agglomeration of βL-Glutamic acid [9–12], and the measurement device enabled modeling the phenomenon with both size and shape information, which would have been difficult with other process characterization tools. Even though the image analysis routine implemented in previous works

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was able to distinguish primary particles and non-convex particles like agglomerates, the need to implement a more accurate and robust image analysis routine was identified. For example, the classification of different primary particles was performed only based on the contour boundary pixels of the particles. But considering the boundary pixels alone, not utilizing the available information in its entirety, would definitely hinder the characterization of complex shapes in a process. Although particle shape characterization by means of supervised and unsupervised learning algorithms is not entirely novel [13], methods for shape categorization based on reconstructed volumetric 3D models have never been applied to the best of our knowledge in the past.

1.1. Contributions

A compact version of the stereoscopic camera setup, ≈5 times smaller than our previous setup, was engineered. A novel calibration procedure for the alignment of the multi-camera setup is proposed, which was missing in our previous works. In contrast to our previous work, the proposed image segmentation algorithm accounts for visual artifacts which finally leads to an improved reliability of measured particle size distributions over time. Furthermore, the novel shape classification of volumetrically 3D reconstructed particles leads to a better shape approximation and segregation of the particles observed by the stereoscopic camera setup. In particular, the more accurate particle shape description allows for a more precise and fine-grained classification of particles which allows for better real-time control of crystallization processes.

1.2. Outline

First, a detailed hardware overview of the measurement device is provided in Section 2. Second, in Section 3, a comprehensive theoretical study of the basic concepts of camera calibration, image analysis, and 3D reconstruction is presented. The measurement device validation and the application of the new particle reconstruction and shape classification is reported in Section 4. Finally, in Section 5, a discussion on the improvements of the measurement device is given along with concluding remarks.

2. Measurement device

A major challenge of imaging-based particle sizing techniques is the dependence of the observed particle size on the orientation of the particle under inspection with respect to the camera. These orientation-related issues can be mitigated by means of a multi-camera setup which provides particle projections from different angles. The previously published stereoscopic imaging setup, henceforth referred to as FTC [8], uses a dual-projection technique capable of merging particle size information provided by two cameras into *n*D PSSDs. This feature yields a more accurate measurement than what single-view setups can provide [2]. However, the major drawback of the FTC is its bulky mechanical design (126 × 126 × 90 cm), making it vulnerable to vibrations during image acquisition. Moreover, the Xenon flashes employed required additional optics to provide collimated light; also, a square flow channel assembled by gluing four sapphire glass windows held by a brass holder was used, making maintenance of the device cumbersome.

Based on the issues described above, a more compact version of the optomechanical setup (80 × 74 × 42 cm) was developed with the goal of overcoming the problems associated with the FTC. The smaller setup described, henceforth referred to as *dual imaging system for crystallization observation* (μ-DISCO), fits into a standard laboratory hood and is less vibration-susceptible during operation. A schematic of the new setup is shown in Fig. 1; it consists of two monochrome CMOS cameras (Point Grey Research, Canada) in an orthogonal configuration with telecentric optics (Opto Engineering, Italy) resulting in an orthographic projection with very low spatial distortions (<0.1%). The camera-lens system provides a field of view (FOV) of 2.41 × 2.02 mm at a nominal magnification of 3.5×. Two high-power, telecentric LED illuminators (Opto Engineering, Italy), which emit collimated chief rays parallel to the optical axis produce high contrast silhouettes of particles passing through the flow channel. The whole setup is mounted on an optical rail cross-construction. Manual XYZ-translation stages (Newport Corporation, USA) that allow high precision alignment of the two cameras, and a rotation stage (Newport Corporation, USA) that allows orienting the parallel illumination beam, are used. A microcontroller (Atmel, USA) running in-house software provides an external trigger signal, which enables a synchronized image acquisition from the cameras.

The introduction of the new cameras allows to operate the μ-DISCO either in a *standard mode*, with a constant low frame rate (1–7 Hz), or in a *burst mode*, with higher frame rates up to 75 Hz. The burst mode is particularly useful for capturing processes with fast dynamics, such as dissolution. In standard mode, the μ-DISCO can be operated either online, that is, the image processing is performed in real-time, or offline, where the image processing is performed after

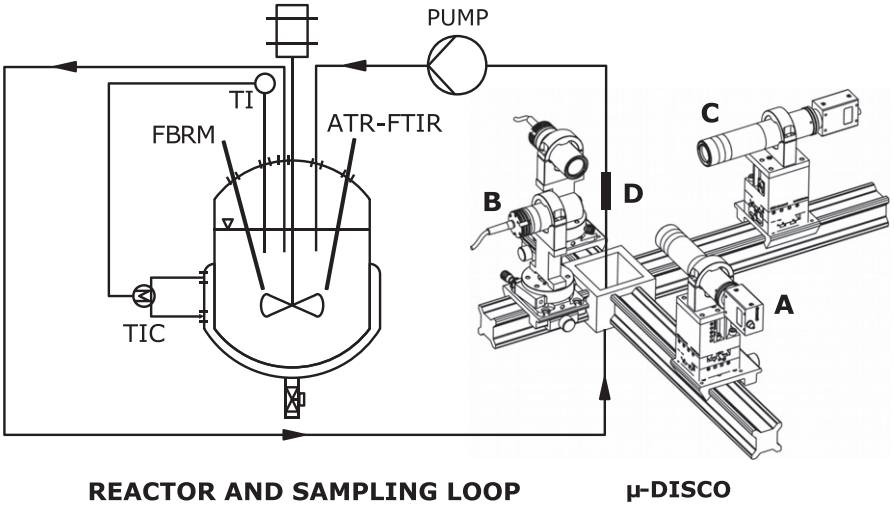

Fig. 1. Schematic of the new dual imaging system for crystallization observation (μ-DISCO). The suspension flowing from the reactor through the flow channel **(D)** is back-light illuminated using two telecentric illuminators **(B)**. The suspension is photographed using two digital cameras **(A)** with telecentric optics **(C)**. The camera, lens and the illuminator system are mounted orthogonally on an optical rail construction.