Application Example: Optimization of Droplet Size and Purity

Motivation

Catalyst particles are produced in a process that starts with liquid–liquid dispersion. Mixing intensity and surfactant dosage are controlled to affect the characteristics of the dispersion. It is known that the properties of the droplets formed are directly reflected in the quality of the final product. A method for analyzing droplets in real process conditions is highly valuable for process development and optimization purposes. The main quality parameter of the dispersion is homogeneous droplet size distribution.

Measurements

A measurement campaign aiming at process optimization was conducted in a pilot-scale reactor in which process conditions similar to manufacturing can be reproduced. The reactor size is about 100L. The process temperature is 60°C and the pressure varies between 2–6 bars. In addition to droplets, the suspension contains gas bubbles as a result of intense mixing. These bubbles are separated from droplets and excluded from the analysis by the Pixact software to prevent them from disturbing the measurements.

The trial program consisted of numerous batches which were measured with the Pixact Droplet Monitoring (PDM) system. It was observed that the characteristics of the droplets vary a lot during the process. This sets high demands on the imaging system and analysis algorithms.

Results

Figure 1 gives an example of droplet size development when the mixing intensity is varied. The figure also shows example images captured by the PDM system at the beginning and end of the measurement. The measurement system enables the continuous monitoring of droplet size distribution.

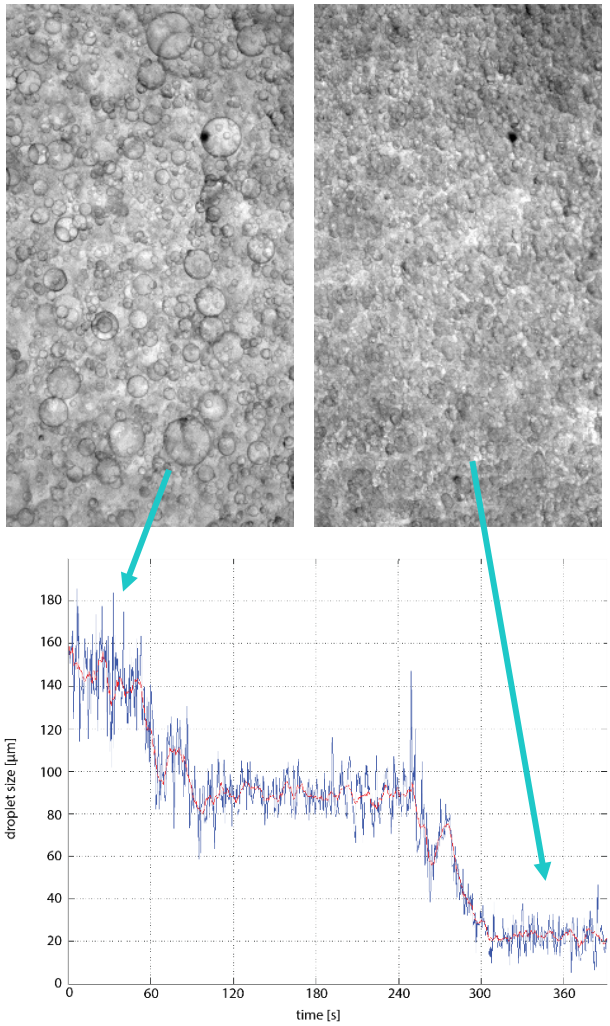

Figure 1. Droplet size trend in varied mixing conditions

Another important quality issue was recognized during the trials. In some batches, with the help of the PDM system, it was observed that droplets and solid particles occur inside the primary droplets. This phenomenon was shown to decrease the quality of the end product. The droplet analysis algorithm of the PDM system was further developed to analyze droplet purity and report the fraction of contaminated droplets in real time.

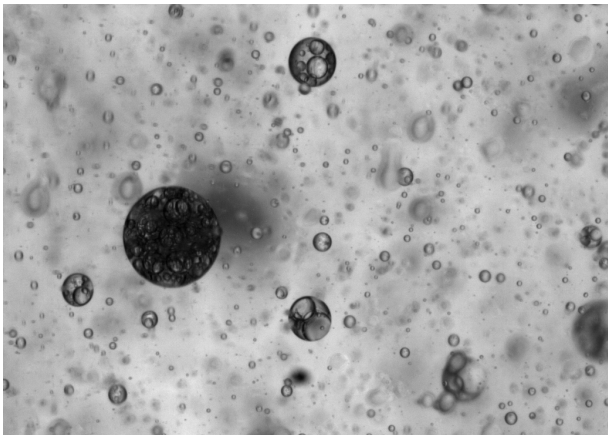

Figure 2. Droplets inside a droplet visualized by the PDM system

Benefits

The PDM system enabled the pilot plant operators and R&D staff to systematically follow the effects of varying process parameters on the properties of the dispersion. Previously, they were not able to gain any information on the size of the droplets during the process: the reactor was a “black box”, and they were only able to analyze the outcome.

In addition to accurate data on droplet characteristics, the Pixact technology provides new insight into reaction kinetics. With the information produced during an eight-week campaign at the customer’s pilot facilities, a remarkable increase in the capacity of the production plant was achieved.

Technical implementation

The PDM system is based on a Pixscope imaging unit (Fig. 3) installed on a DN65 side flange of a pilot-scale reactor. As the reactor is located in EX space, an ATEX-certified version of the PDM system was used. Only the imaging and main units are located in the field. The operator station (analysis computer and monitor) is located in the control room in another building. The connection to the imaging unit is established with an optical fiber. A schematic layout of the measurement system is presented in Figure 4.

A summary of the specifications of the measurement system is presented in the table below.

Probe head	Pixscope 64-100 / DN65
Material	Stainless steel AISI316L
Gaskets	FPM
Windows	Sapphire
Imaging resolution	1.7 µm/pix
Image area	4.4 mm x 3.2 mm
Optimal measurement range	10-500 µm
Software	Pixact Droplet Monitoring

Figure 3. Pixscope DN65 probe

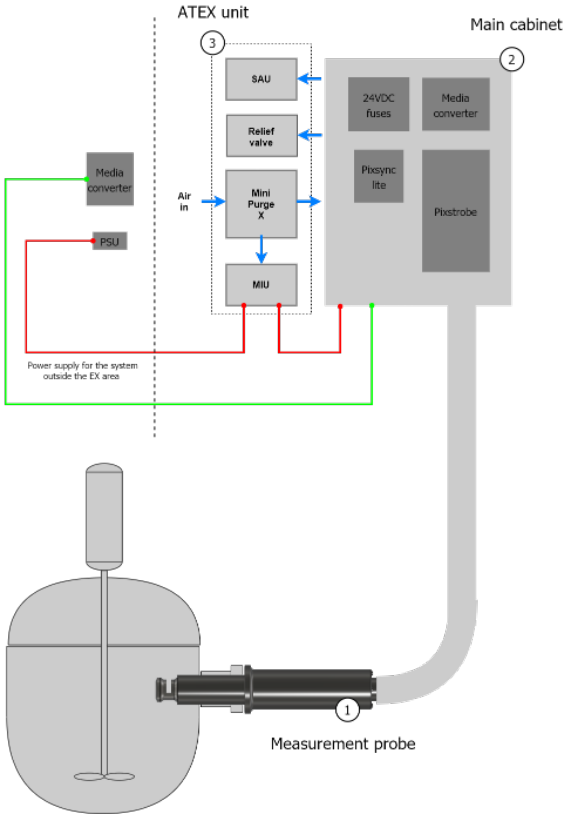

Figure 4. Layout and connection diagram of the measurement system