Particulate Size Analysis for Process Optimization

Particles, crystals, and droplets occur in many chemical processes, across a range of industries, and often pose challenges for scientists and engineers who are tasked with optimizing product quality and process efficiency. Characterizing particle properties effectively, in particular particle size and count, allows processing problems to be solved and product quality to be improved. Historically, scientists have relied on off-line particle size analyzers, such as laser diffraction or sieving to perform this type of characterization. But in recent years, newer technologies have emerged, which describe particle size and count in real time, as particles naturally exist in process. In process measurement of particles can reduce the error associated with offline sampling, and can provide continuous information about how particles behave under changing process conditions, allowing scientists to understand and optimize difficult processes using evidence-based methods. This article will introduce some of the most common in process particle measurement approaches and how they can be deployed for the effective delivery of high quality particle products.

Figure 1. Particle size and count can influence both product quality as well as process efficiency.

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1 Introduction

Particles and droplets are present in the vast majority of manufacturing processes and their final products. The proper control of particle size, shape and count is often a critical factor in final product quality and can greatly influence process efficiency (Figure 1).

For example, the effectiveness of medicines used to treat lung diseases has been shown to depend heavily on particle size, with large particles exhibiting poorer penetration into airways. It has also been reported that particulate process plants take longer to start up, and are less likely to achieve desired production rates, versus those processing liquids or gases. Multiphase systems involving combinations of particles, droplets, and bubbles result in additional complexity and magnify the challenge of understanding, optimizing, and controlling the processes used to produce, modify, or separate them.

The determination of optimized process parameters is critical to ensure the correct particle size and count can be obtained consistently. In a process such as crystallization, the cooling rate chosen will directly influence final crystal size, with faster cooling rates typically delivering smaller sizes. In emulsification processes, it has been shown that mixing intensity must be controlled in order to obtain the desired droplet size distribution. In the flocculation of fiber cement particles, high molecular weight polymer flocculants have been shown to increase aggregation resulting in larger and stronger flocs.

To successfully deliver particulate products to the market, scientists must develop a comprehensive understanding of how process parameters affect particle size and count. They must choose operating conditions that will deliver particles with the required attributes consistently and cost effectively. In order to do so, reliable particle characterization methods are needed, and the results obtained from these methods must be easily related to the process parameters that govern the outcome of the process.
2 Traditional Particle Size Analysis

Traditional Particle Size Analysis (PSA) using an offline analyzer is a powerful and widely used technique for the measurement of particle size in quality control (QC) labs. Examples of traditional particle size analysis techniques include sieving, laser diffraction, dynamic light scattering, and electrozone sensing. This approach allows QC laboratories to check the specification of particles at the end of a process against a set specification and identify deviations from the required particle properties. In order to obtain useful results from traditional particle size analyzers, it may be useful to consider the following points:

Sampling and Sample Preparation
A successful laboratory analysis of particle and droplet systems requires the removal of a representative sample from the process, and the preparation of this sample for analysis. Most PSA techniques have strict constraints on the range of concentration, size, and shape of particles that can be measured accurately. This sample preparation procedure often involves multiple steps necessary to meet these measurement constraints and can employ methods such as filtration, rinsing, drying, subsampling, resuspension, surfactant addition, dilution, and sonication. However, it is quite possible that these steps may significantly alter the particles or droplets of interest. Even with the utmost care and precision in the sampling and sample preparation methods, the actual particles that are analyzed may be significantly different from the particles that were initially present in the process vessel (Figure 2). For this reason, particles must be sampled in such a way as to minimize the possibility that change might occur during the removal, preparation, or measurement phases of the procedure.  

Consideration of Particle Shape
Many particle size analyzers assume particles are spherical to allow simplified models to be applied in order to report consistent results. A sphere is the simplest particle shape in the sense that one number, the diameter, describes the particle completely. Often however, particles are non-spherical (Figure 3), and changing particle shape can be even more important than size in determining bulk solids properties, such as flowability, and filterability. Scientists must take care to understand how particle shape influences a traditional particle size analyzer, and in cases where particles are known to be non-spherical, it is important to take this into account when analyzing results.

Figure 2. Mannitol crystals compared in a. process using real time microscopy and b. offline using a standard light microscope. Sampling and preparation for microscope analysis have resulted in significant breakage and delicate dendritic structures observed in process go undetected.
The particle size of non-spherical particles is often reported using an equivalent diameter; which is the diameter of a spherical particle which will give identical geometric, optical, electrical or aerodynamic behavior to that of the particle (non-spherical) being examined. In Figure 4, particles with different shapes but equivalent volume are depicted. If particle size is reported for each of these particles based on volume, then the same particle size will be reported in each case. If particle size is reported based on sieve diameter (during sieving, a particle hits the sieve mesh until it passes with its smallest projection screen through an aperture) then different particle sizes are reported. Since changes in particle shape can influence process and product quality, care should be taken to determine how shape influences particle size analysis results, and if possible to determine particle shape using a technique such as imaging.

Another important consideration is that particle systems are composed of a population of particles with different sizes and shapes. Many traditional particle size analyzers report a particle size distribution, from which an average (typically, a mean or median) is calculated and reported. Care must be taken to consider how particle count at the fine and coarse tails of such a distribution influence the reported particle size.

### Time Delay

Since most particle process streams operate at a solids loading much higher that anything traditional particle size analyzers can handle, careful sample preparation is needed for effective measurement. It is virtually impossible to apply PSA measurements directly in a process. This means that traditional offline particle size analyzers are not easily implemented to obtain real-time information, as process parameters are varying. In order to do so, slip streams with automated dilution and preparation systems would be required. However, even with a successful setup, the reliability of such measurements would be questionable given the likelihood of particles changing dramatically during diversion, preparation and analysis.
In order to obtain continuous information about how particle size relates to process parameters, samples would have to be manually extracted and analyzed on the fly. This approach is challenging from a cost perspective and may expose those taking the samples regularly to an unacceptable level of risk—especially for processes at elevated temperatures and pressures with toxic or explosive slurries and solvents. The inevitable time delay between sampling and the receipt of results for traditional PSA makes them extremely difficult to implement for any kind of real-time measurement, and makes them unsuitable for monitoring processes continuously as they change over time.

**Summarizing Traditional Particle Size Analysis**

Offline particle size analysis is a powerful and widely used technique for the measurement of particle size, and for comparison with a set specification in QC. With care, traditional particle size analysis can be used to identify variations in product quality, and can be used to ensure that products meet the specifications required by producers, their customers, and regulators who oversee the quality of products reaching the public.

However, traditional particle size analysis does not lend itself well to characterizing particles continuously as process parameters change, and for this reason they are not especially suited to the task of process optimization. It is extremely difficult to rely on a single offline sample, no matter how reliable the data obtained, in order to completely understand particle behavior from the beginning until the end of a process. In order to develop truly effective process understanding and to translate this into meaningful improvements for the process, continuous measurements are needed that characterize particles in real-time as they naturally exist in the process. With this information, particle mechanisms such as growth, breakage, and agglomeration can be directly observed, the influence of process parameters on the system can be determined, and an optimized route to the desired particle properties can be identified and implemented quickly.

### 3 In Process Particle Measurement

In-process particle measurement typically relies on inserting a probe-based instrument into a process stream for direct measurement of particles as they naturally exist in the process (Figure 6). This type of measurement occurs at full process concentrations and does not require sampling. Typically, probes can be applied across a range of scales and installation environments, ranging from small-scale laboratory reactors to full-scale production pipelines.

**Study Particle Size and Count Over Time**

Typically, an in-process particle measurement is taken every few seconds, allowing discrete distributions to be recorded at user-defined intervals. Statistics from each distribution can then be trended over time allowing scientists to monitor process trajectory in real-time (Figure 7). By monitoring particles in process and in real-time, it is straightforward to determine (1) when particle size and count start changing; (2) when particle size and count stop changing; (3) the rate at which particles change; (4) the degree to which particles change. With this information, scientists can develop a much deeper understanding of their processes compared to the case where they must rely on a single particle size analysis result from a single point in space and time.

![Figure 6. In process particle measurement instruments using focused beam reflectance measurement technology (see Appendix) implemented in lab and production settings](image-url)
Understand the impact of process parameters on particles

An in-process approach to particle measurement differs significantly from the role traditional particle size analysis play in the characterization of particles. In process measurement takes place directly in the vessel or pipeline, while the particles are changing, rather than in the quality control lab and results are immediately related to dynamic process conditions, rather than to a predetermined particle size specification. By combining relevant process parameter information with in-process particle measurements it is possible to quickly obtain evidence that can be used to optimize processes with scientific rigor (Figure 8).

Where did the small particles come from?

Figure 7a. Discrete distributions can be recorded in real time at user defined intervals

Figure 7b. Statistics such as particle count in individual sizes classes can be trended continuously in real time

Figure 7c. Trended statistics provide key information regarding changes to particle size and count including: 1. When does the process start? 2. What is the rate of change? 3. When does the process end? 4. What is the degree of change?

Figure 8. In process particle measurement identifies the formation of small particles at the same time the rate of cooling increases
Choose Parameters to Deliver the Correct Particles
By directly monitoring the impact of process parameters on particle size and count it is possible to reliably determine the process parameters needed to target a specific set of particle attributes. In Figure 9 the impact of agitation rate on droplet size is clearly shown allowing scientists to readily choose a set of operating conditions that will deliver the desired droplet size.

Monitor and Correct Process Deviations
By monitoring particle size in real time directly in the process it is possible to identify process deviations and take corrective action to minimize the impact of the upset. In figure 10 a continuous process is being monitored where particle size must be kept within tight specifications. Here it should be noted the specification that is set using the in-process instrument is not necessarily the same as the specification set using the traditional particle size analyzer in the QC lab. Here the in process particle measurement instrument is simply identifying a dramatic process upset and can support troubleshooting the problem before implementing a corrective action that will bring the process back into specification.

Figure 8. In process particle measurement shows influence of agitation intensity on droplet size.

Figure 10. Using in process measurement to identify a process upset during a continuously operating process and take corrective action.
For batch processes in process particle measurement can support the reduction of batch time by identifying when a process reaches steady state. In Figure 11 most of the process changes occur during the first two hours of the process – however in process particle measurement indicates that particles do not change for the remaining 10 hours of the batch. This is a good indicator that batch time could be reduced – and that a sample for offline quality control should be taken soon after the initial 2 hour period, where most change occurs.

**Summarizing In Process Particle Measurement**

In-process measurement of particles is suited particularly well to developing process understanding for complex particle systems and for determining the appropriate parameters needed to deliver particles with the correct properties. In process particle measurement also complements traditional particle size analysis by supporting quality control efforts through the identification and rectification or process upsets during production.

- Avoid errors associated with non-representative sampling
- Avoid physical changes to the particle resulting from sampling, transport, storage, sample preparation, and flow through the off-line measurement instrument
- Obtain continuous and real-time information about the particle system as process parameters are changing
- Characterize particles where sampling is challenging due to temperature, pressure, or toxicity
- Directly observe the impact of disturbances and intentional process upsets

![Figure 10](image-url)  
*Figure 10:* A batch process where the final 10 hours of the process does not exhibit any change in particle size or count
4 Conclusions

Particle size and count are important to characterize effectively for the successful development, transfer, and operation of processes in numerous industries. Traditional particle size analyzers are used in the quality control laboratory to measure particle properties with accuracy, however care must be taken to prepare the sample to allow for a consistent measurement. The time delay and potential for particle changes between sampling and analysis make the traditional particle size analysis approach challenging for process optimization and improvement.

In process measurement instruments offer an opportunity to track how particle size and count change directly in the process in real time. By understanding how particles behave from the beginning until the end of a process, and by comparing particle changes to process parameters, scientists can develop a deep understanding of particle systems. This allows processes to be optimized using evidence based methods and for troubleshooting to be executed during production.

In process particle measurement complements traditional particle size analysis by providing extra information about how particles actually behave naturally in process. If a quality control lab reports a deviation from specification in process particle measurement can be used to perform root cause analysis. Likewise, in process particle measurement can predict when a process will move out of specification and can help identify when a sample should be taken from a process for offline analysis and quality verification.

By combining in process particle measurement for understanding, optimizing and troubleshooting processes with traditional particle size analysis for quality control scientists can develop particle processes with higher quality, in less time at a lower total cost.
5 Appendix A: ParticleTrack with FBRM®
(Focused Beam Reflectance Measurement)

ParticleTrack™ with Focused Beam Reflectance Measurement® (FBRM) technology is a probe-based instrument that is inserted directly into processes to track changing particle size and count in real time at full process concentrations. Particles, particle structures, and droplets are monitored continuously, as experimental conditions vary, providing scientists with the evidence required to deliver consistent particles with the required attributes.

How does ParticleTrack work?

The probe is inserted directly into process streams, at an angle, to ensure particles can flow easily across the probe window where the measurement takes place. A laser beam is launched down the probe tube through a set of optics and focused to a tight beam spot at the sapphire window. The optics rotate at a fixed speed (typically 2 m/s) resulting in the beam spot rapidly scanning across particles as they flow past the window.

As the focused beam scans across the particle system, individual particles or particle structures will backscatter the laser light to the detector. These distinct pulses of backscattered light are detected, counted, and the duration of each pulse is multiplied by the scan speed to calculate the distance across each particle.

This distance is defined as the chord length, a fundamental measurement of the particle related to the particle size. Typically thousands of particles are counted and measured per second, allowing a precise and highly sensitive chord length distribution to be reported in real time.

The chord length distribution tracks how particle size and count change from the beginning, until the end of a process. Statistics from each chord length distribution, such as counts in fine and coarse size classes, can be trended over time.
6 Appendix B: ParticleView with PVM Technology

ParticleView V19 with PVM® technology is a probe-based instrument that visualizes particles and particle mechanisms in real time. High resolution images are continuously captured without the need for sampling or manual offline analysis. A process trend, sensitive to changes in particle size and concentration, is automatically combined with the most relevant images providing scientists with comprehensive process understanding.

www.mt.com/ParticleView

How does ParticleView work?
ParticleView uses a high resolution camera and internal illumination source to obtain images even in dark and concentrated suspensions or emulsions. With no calibration needed and easy data interpretation, ParticleView quickly provides critical knowledge of crystal, particle, and droplet behavior.

What is RBI?
ParticleView V19 with iC PVM uses information from every image that is collected to calculate an innovative process analytical trend called “Relative Backscatter Index (RBI). RBI is a measure of the overall reflectivity of a particle system and indicates how particle size, shape, and concentration is changing over time.

RBI is used to understand how changing process parameters affect process performance and combined with high resolution images provides comprehensive process understanding.
7 References