THE PRACTICALITIES AND BENEFITS OF INLINE TECHNOLOGY FOR MONITORING MIXING

This article examines the practicalities and benefits of inline technology for monitoring mixing processes, focusing on the technique of drag force flow measurement, which provides highly sensitive, real-time data. Experimental studies illustrate the capabilities of this process monitoring technique for a range of mixing and blending applications.

John Yin, Freeman Technologies
Dr. Valery Sheverev, Lenterra

The need to blend multiple powders or mix powders with liquids is commonplace across the bulk powder processing industries. The associated processes that come with blending and mixing are energy-intensive and have the potential to damage primary particles depending on the particles' friability and the processing conditions applied. Just enough processing to reach a satisfactory endpoint is highly desirable from the perspective of minimizing variable costs, as minimal processing will mitigate the negative impacts of overprocessing and will maximize equipment usage (material throughput). Achieving this goal of a satisfactory endpoint relies on the timely detection of homogeneity, and one of the best ways to do that is with real-time process monitoring.

The benefits of real-time monitoring

Over recent decades, real-time process monitoring using inline instrumentation has become increasingly prevalent across the processing industries, and there are sound reasons for this trend. For starters, such measurements typically involve minimal process disruption, if any at all, since they eliminate sample-removal requirements. Measuring materials *in situ*, meaning within the process, captures data for a much larger proportion of the process stream than is feasible via discrete sampling and offline analysis. Additionally, inline systems measure materials under process conditions, which is particularly beneficial for powders, since powder properties are dependent on the stress and strain rates applied to the material. For these reasons, inline powder measurements can be significantly more representative than offline and at-line testing.

Above all, the greatest benefit of inline technology is its ability to deliver better representative data in real time. Switching to real-time measurements transforms informational flow from a single analysis every hour or two to a continuous data stream tracking the process minute by minute. Offline analyses are not only infrequent but also temporally offset from the process due to delays introduced by sampling, transferring the sample to the lab, analyzing the sample, and delivering the results. These delays significantly complicate the challenge of maintaining optimal operations. In contrast, with real-time data it's possible to:

- immediately detect deviations from a given process setpoint to consistently maintain the process in the preferred operating range,
- reduce overprocessing by stopping at a precisely defined, optimal endpoint,
- and minimize any downtime associated with waiting for results, such as with a batch release, thereby maximizing unit throughput.

Understanding the generalized benefits of inline processing can be helpful in making the most of your mixing and blending processes. However, it's also important to be aware of the downsides of relying solely on offline measurement techniques to find your materials' homogeneity specifications.

The reality of offline measurements' impact on the process

The goal of mixing or blending is typically to process materials to an acceptable level of homogeneity, which happens at a very specific period in the process. Processing time is influenced by the physical properties of the particles or powders, including particle size, morphology, surface texture, and cohesion. Processing time can also be controlled by manipulating critical processing parameters, including the geometry and design of the vessel and impeller, and, once these are fixed, impeller speed. In the absence of inline measurements, samples are extracted from the process and analyzed offline to assess the degree of homogeneity.

Offline measurements provide limited opportunity to closely track the blending process toward completion and/or rapidly detect any mechanical failure of the equipment's impeller. Operators will feel the urge to safeguard the homogeneity specifications due to the time delays associated with offline analysis. Operators may also experience a general lack of confidence in being able to exert close control over the process. As a result, executing the process using offline measurements is highly likely to cause routine overmixing. This "safety margin" carries the unintended costs of wasted energy, compromised throughput, and an increased risk of particle damage, which could impact product quality. Inline technology that precisely and instantaneously detects the endpoint of a mixing process eliminates these issues, resulting in considerable economic return.

Assessing requirements for inline powder characterization technology

To successfully deliver the benefits of real-time measurement, inline technology must answer to a demanding set of requirements. Process environment reliability and suitability are critical for any continuous monitoring technology, but for bulk powder characterization, relevance and sensitivity are of particular importance because of their uniqueness to the bulk powder process. The sampling technique must measure a property that correlates process or material performance with sufficient repeatability and sensitivity to detect subtle but crucial differences. These issues similarly impact the value of offline powder testing methods.

Relevance. Powders are routinely tested in many ways across the various powder processing industries, a common goal being to rank or quantify material's flowability. Traditional methods for assessing flowability include measuring the material's angle of repose, a manual technique that involves measuring the angle at which a powder settles when poured onto a flat surface. Though simple measurement methods can differentiate powders to some degree, the relevance of the resulting data to a specific process may be unclear. For example, if Powders A and B each have an angle of repose of 34 and 41 degrees, respectively, what does that mean in terms of their relative blending performance? Powder testing methods vary significantly in terms of their ability to generate data that correlates with performance in a process, but without such correlations, the data is of limited value.

Sensitivity. Many offline powder testing methods suffer from poor repeatability often because they are manual or have poorly defined protocols or methodologies. Poor repeatability erodes the sensitivity of a method, but techniques are inherently different in this respect. Sensitivity is a function of the variable being measured, the instrumentation used, and, in some instances, sample type. For example, current shear cells are moderately sensitive for more cohesive materials because the absolute value of the measurements being made is relatively large but much less sensitive for more free-flowing powders, which generate lower values of shear stress.

Judging inline solutions for bulk powder characterization against the criteria of relevance and sensitivity is vital when it comes to robustly determining their potential to improve your process.

Inline powder characterization by DFF measurement

An inline drag force flow (DFF) sensor is a relatively new technology for real-time bulk powder characterization that measures the local forces associated with the flow of powders, granules, or wet masses within a process. This technology uses fiber-optical strain gauges that offer integrated temperature compensation and measurement and are well-suited to the process environment. While there are alternative inline technologies available, the DFF sensor is of particular interest because it has been shown to produce data that correlates closely with offline dynamic data, which has widespread applicability to the bulk powder processing industries. As you'll read in this article, the DFF sensor has proven relevance in mixing and blending applications.

The DFF sensor, or pin, as shown in Figure 1, has a fine, needle-like structure with a hollow core and an outer diameter of approximately 1 to 4 millimeters. The size of the sensor results in minimal flow disruption when the sensor is inserted into a process. Mounted opposite one another on the inner surface of the sensor are two fiber-optical strain gauges or fiber Bragg

FIGURE 1

A drag force flow sensor deflects in response to the flow of process materials.

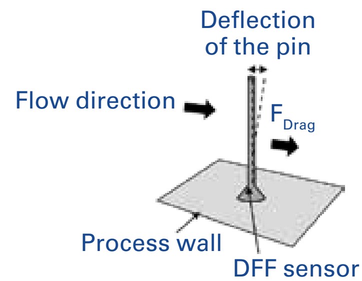
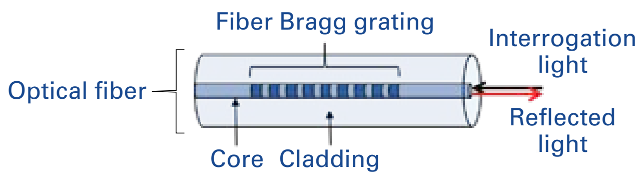


FIGURE 2

A fiber Bragg grating is a short segment or structure of varying refractive index within an optical fiber.



gratings (FBGs). An FBG, shown in Figure 2, is a short segment or structure of varying refractive index within the core of an optical fiber; cladding surrounds the FBG to give it mechanical integrity. A critical characteristic of an FBG is that the application of flow force induces a shift in the wavelength of interrogation light that it reflects.

Materials flowing past the sensor cause a deflection of the pin (sensor), which flexes from its anchored base as demonstrated previously in Figure 1. The magnitude of this deflection correlates with the local flow force, referred to as F_{Drag} in Figure 1, which is associated with powder movement within the process. The FBG on one interior wall is subject to tensile forces while the FBG diametrically opposite undergoes compression, giving rise to the relative spectral shifts, labeled as $\Delta\lambda$ in Figure 3. The wavelength of light reflected by an FBG can also be shifted by temperature changes, but both FBGs would be equally affected where this occurs. Spectral shifts associated with force and temperature are, therefore, easily deconvoluted, allowing the sensor to self-calibrate for temperature. The force associated with the movement of particulate flow is measured precisely in real time by applying a light source (interrogation light) and tracking shifts in the reflected light or resonant wavelength.

DFF sensors offer multiple advantages for bulk powder characterization when judged against the criteria discussed earlier. These include:

- a sensing mechanism well-suited to the process environment that is unaffected by electromagnetic interference and doesn't present an ignition hazard,
- high-frequency measurement (up to 500 hertz) at high resolution (less than 10 micronewtons), making it possible to precisely and sensitively track even rapidly changing processes,
- an enclosed, stainless steel construction that offers excellent resistance to a wide range of materials, boosting reliability,
- and turnkey operation when integrated with an optical interrogator and associated software.

Crucially, the data generated by DFF sensors has also been found to correlate closely with dynamic powder flow properties.[1] Dynamic testing is a high-sensitivity, offline method with proven relevance for a wide range of industrial processes from fluidization to granulation.[2] Correlations between DFF and dynamic data highlight the relevance of DFF technology and the potential to transfer valuable specifications already in place directly into the processing environment for real-time monitoring. Dynamic testing is an established method for assessing the mixing and blending of powders. The following case studies illustrate the application of inline DFF sensor technology in comparable applications.

Case Study 1: Monitoring mixing behavior and the influence of particle properties

Batch mixing studies were carried out using four grain samples: couscous (CC), green lentils (GL), pearl barley (PB), and long-grain rice (LG). Baseline data was gathered for 20 seconds with the mixer off, and then samples were mixed for 120 seconds. The mixing,

FIGURE 3

In a drag force flow sensor, flow force induces opposing shifts in the wavelength of light reflected by the two fiber Bragg gratings (left graph) while a change in temperature causes an identical spectral shift for both fiber Bragg gratings (middle and right graphs).

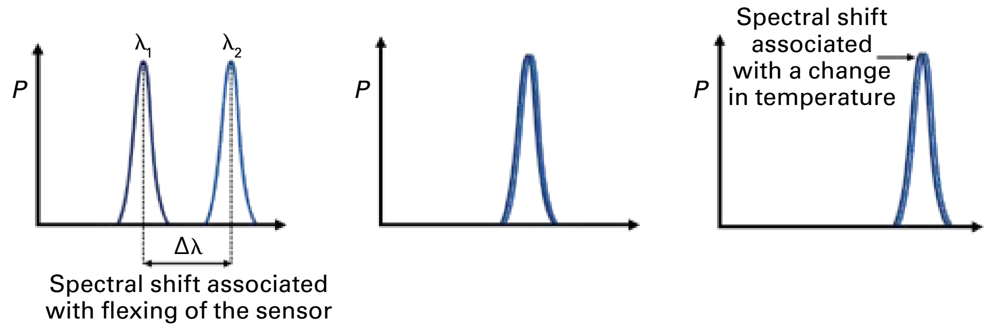


FIGURE 4

Real-time force pulse magnitude data exhibits high repeatability and clearly differentiates the four grains.

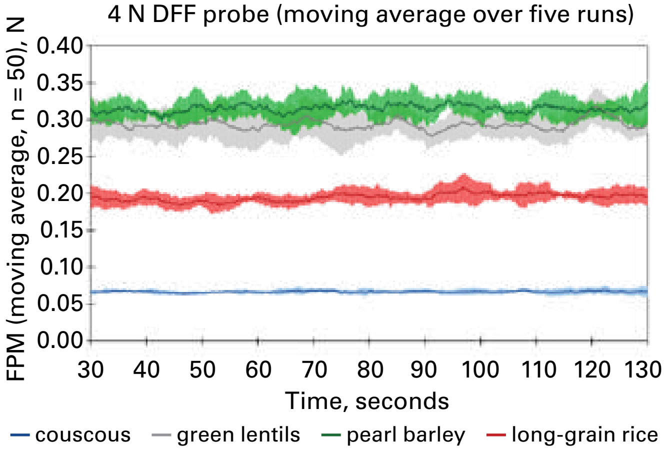


TABLE I

Blend compositions for a series of trials mixing grains with large and small particles in known volumetric ratios (%v/v).

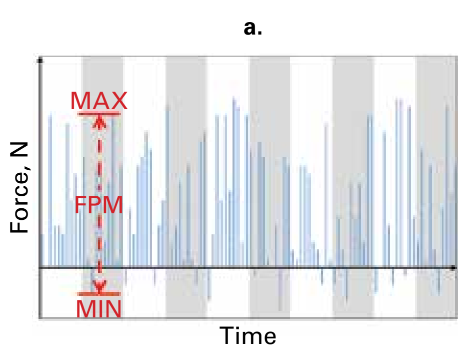
Test Number	LP Content, %v/v	SP Content, %v/v
1	0	100
2	25	75
3	50	50
4	75	25
5	100	0

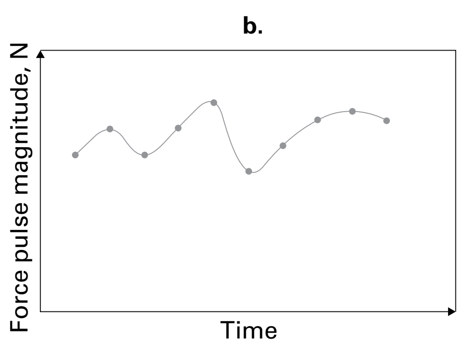
which was done with a simple impeller, was monitored with a 4-Newton (N) DFF sensor with five repeat measurements made for each sample.

Figure 4 shows measured data for each of the four samples expressed in the form of moving average force pulse magnitude (FPM) measurements. Figure 5 illustrates how FPM data is derived from raw DFF measurements, which are associated with the impact of individual particles on the sensor. The FPM is the difference between the minimum and maximum

FIGURE 5

Raw flow force data measured by a drag force flow sensor (a.) is usefully converted into force pulse magnitude values for process monitoring (b.).





force in a given data array, the data associated with a time period selected in reference to the process. FPM expresses the magnitude of the force variation and, unlike the raw data, is always positive, making it highly suitable for process monitoring. With a parameter that is always positive, monitoring simply involves comparing magnitude or difference relative to setpoint. When a parameter can be both positive and negative, monitoring becomes more complex. For process monitoring and control, a signal that stays positive is therefore easier from the perspective of formulating control decisions, whether they be manual or automated. Furthermore, as a differential measurement, FPM values are unaffected by drift in the raw signal.

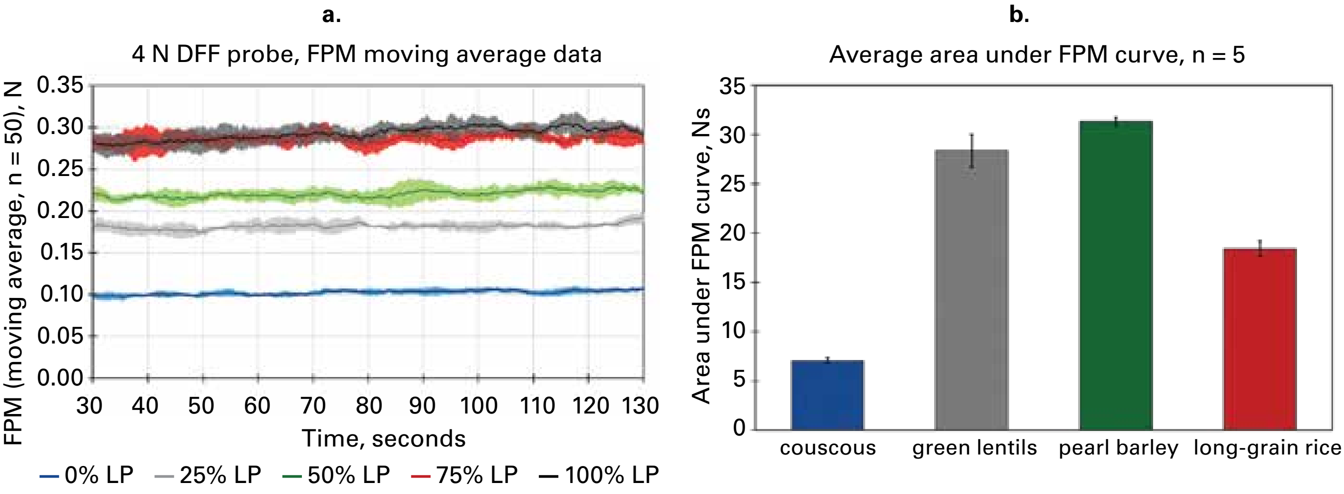
The FPM data shows high repeatability across the five runs and indicates uniformity within the blended batches. All four grains are clearly differentiated on Figure 4, with the couscous, which has a spherical shape and the smallest particle size, generating the lowest FPM values. Particle size, shape, and density, along with the cohesivity of the grains, are all likely to contribute to the FPM rankings exhibited.

In an extension of this trial, two new grains with small particles (SP) and large particles (LP) were blended alone and as binary mixtures, as shown in Table I. Blending conditions were the same as for the earlier trial with baseline data gathered for 20 seconds and samples then mixed for 120 seconds; five repeat measurements were again made for each sample.

Again, these trials produced highly repeatable data and evidence of blend uniformity, as shown in Figure 6, graph a. There is also a clear, positive, nonlinear trend between the percentage of large particles in the blend and the magnitude of the FPM signal, as summa-

FIGURE 6

Force pulse magnitude data exhibits a positive, nonlinear trend with large particle content (%LP) and effectively differentiates the blends.



rized by the integrated area under the FPM curve data in Figure 6, graph b.

In summary, this study shows that the DFF sensor effectively differentiates a range of grains and robustly detects the nonlinear impact of varying the composition of grain blends with large and small particles.

Case Study 2: Monitoring liquid dispersion in a vertical twin-shaft mixer

In the second study, a DFF sensor was used to monitor the dispersion of a liquid component in a powdered food product being mixed in a vertical twin-shaft mixer, as shown in Figure 7. Repeat measurements of the dispersion process were carried out with the liquid addition rate and the total liquid amount kept constant between the two runs. While physical changes in the process were monitored in real time using the DFF sensor, samples were also extracted at two locations, as shown in Figure 7, for offline analysis. The liquid component contained appreciable levels of sodium chloride (NaCl), and therefore, its distribution through the powder could be efficiently measured offline by potentiometric titration. Potentiometric titration is a chemical method of analysis in which the material's homogeneity is monitored with an indicator electrode; as testing methods go, it is generally not that expensive and is reliable and readily available.

Moving average FPM data for the two runs shows highly repeatable trends, as indicated in Figure 8. Liquid addition causes an increase in FPM as particles coalesce and capillary bonds strengthen between particles, but FPM values then decrease rapidly as the liquid disperses through the powder. A clear plateau of FPM values establishes after approximately 25 seconds of blending, indicating that mixing for this length of time is sufficient to produce homogeneity in the powder. The potentiometric titration offline analysis shows similar trends. Sampling location 2, which is closer to the liquid injection nozzle, initially has a higher sodium chloride concentration than Sampling location 1, but from approximately 24 seconds and onward, the two sampling points generate closely similar values that change minimally with further mixing. The two monitoring techniques indicate almost identical times for even liquid dispersion to the point of blend homogeneity.

Therefore, the DFF sensor closely replicates an established offline technique, generating comparable data in real time with no requirement to disturb the process to determine progress toward the desired endpoint.

FIGURE 7

A vertical twin-shaft mixer is set up for dispersing liquid in a powdered food product. The drag force flow sensor is installed directly in the mixer.

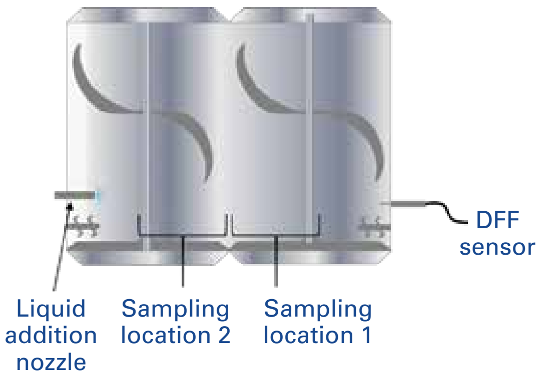
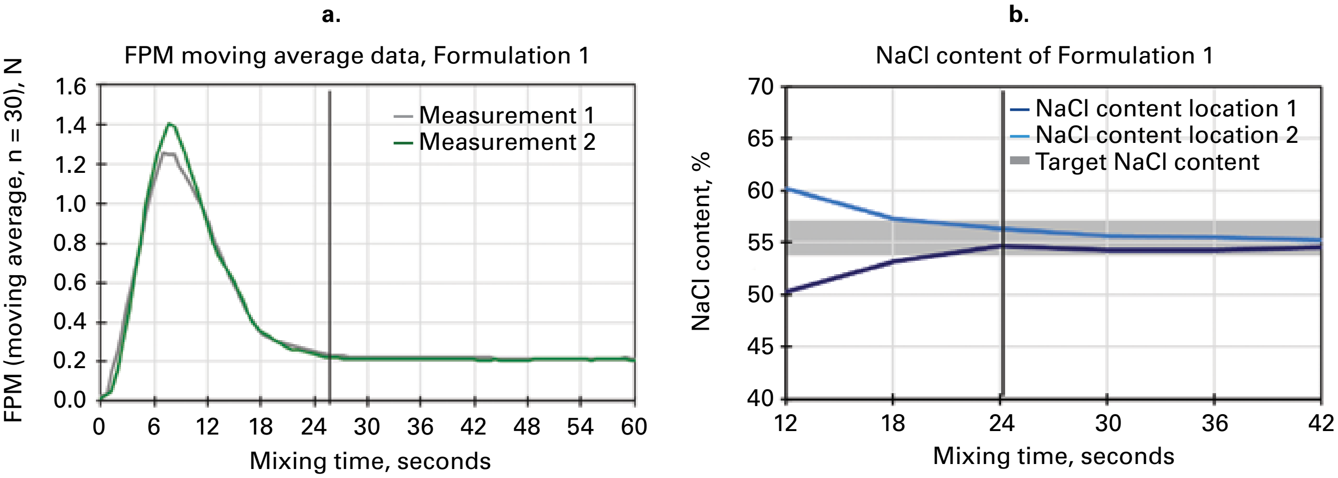


FIGURE 8

There is close agreement between the drag force flow and potentiometric titration data with both indicating that the blend reaches a state of homogeneity after approximately 24 to 25 seconds of mixing.

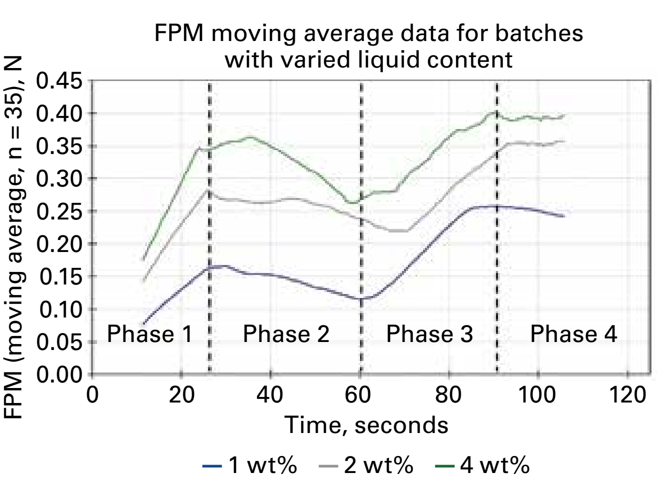


Case Study 3: Investigating the impact of liquid addition in a vertical single-shaft mixer
In the final study, a DFF sensor was used to investigate the impact of adding different quantities of liquid to a powdered food blend. Measurements were made with a vertical single-shaft mixer, adding liquid to a level of 1, 2, and 4 weight percentage (wt%). The liquid addition rate was adjusted to maintain a consistent liquid addition time across all three measurements.

The FPM data displayed in Figure 9 for all three liquid concentrations shows similar trends and four clear phases of the wet massing process. At the start of liquid addition, Phase 1, FPM values rise. This can be attributed to the capillary bonds growing in strength

FIGURE 9

Real-time force pulse magnitude data closely tracks liquid addition, clearly differentiating the mixtures and identifying different phases of the wet massing process.



and particles beginning to coalesce, as observed in Case Study 2. Liquid addition continues during Phase 2, but at this point, the choppers in the mixer have been switched on. These break down larger agglomerates, reducing particle size and decreasing FPM. Phase 3 coincides with switching off the choppers, which allows the particles to further grow and densify as mixing continues. By the beginning of Phase 4, liquid addition has ceased, and more friable agglomerates are being broken down, limiting the potential for any further increase in FPM; the process has essentially reached its endpoint with little further benefit associated with additional processing. Indeed, there is some evidence of a slight reduction in FPM, particularly with the low water-content sample, a trend associated with agglomeration breakdown.

This data clearly shows the DFF sensor's ability to differentiate the impact of varying levels of water addition and to elucidate different phases of the water-addition process.

It's worth noting that real-time process monitoring using inline instrumentation isn't meant to replace offline measurements completely. Inline instrumentation, such as DFF sensors, should be tested via experiments, such as the ones presented here, until the preferred inline instrument has been proven to match offline measurements. At that point, the inline instrumentation can be used as the primary method for monitoring processes with the offline tool being used as necessary, particularly with final quality check product specifications, which are subject to routine offline checks.

In conclusion

Inline bulk powder characterization technology can play an important role in supporting the optimization of mixing and blending processes. Real-time monitoring of these processes enables precise detection of the point at which homogeneity is reached, offering opportunities to eliminate the inefficiencies and economic penalties associated with overmixing.

The data presented in this article illustrates the potential of DFF measurements within this context, highlighting the technique's suitability for monitoring dry powder blending and wet mass mixing processes. DFF sensors are robust instruments capable of detecting small differences in the properties of process materials with a high degree of sensitivity. These results show the sensor's ability to identify different phases of a mixing process and detect the point at which a blend becomes homogeneous. The agreement between FPM and offline potentiometric titration measurements points to the inline measurement technique's suitability for transferring established offline specification measurements into the process environment, as illustrated by the reported correlations between DFF data and dynamic powder properties. These findings underline the relevance and value of DFF measurement as a technique for real-time powder process monitoring. **PBE**

References

1. A.S. Narang, "Process analytical technology for high shear wet granulation: Wet mass consistency reported by in-line drag flow force sensor is consistent with powder rheology by at-line FT4 powder rheometer," *Journal of Pharmaceutical Sciences*, January 2016, pages 185-187.
2. J. Clayton, "Reviewing current practice in powder testing," *Organics Process Research & Development*, Vol. 19, No. 1, pages 102-109.

For further reading

Find more information on this topic in articles listed under "Mixing and blending" in *Powder and Bulk Engineering*'s article index in the December 2019 issue or the article archive on *PBE*'s website, www.powderbulk.com.

John Yin (john.yin@freemantech.co.uk, 718-358-2058) is the US product manager for Freeman Technology and provides comprehensive support and education for new and existing users of Freeman's technology. John regularly presents at conferences and workshops throughout the US on the subject of powder rheology, helping industry and academia to further understand powder behavior. John earned a bachelor's degree in engineering chemistry from Stony Brook University in 2001.

Freeman Technology
Tewkesbury, England
732-829-8345
www.freemantech.co.uk

Dr. Valery Sheverev (sheverev@lenterra.com, 973-623-0755) is the president of Lenterra Inc. Valery founded Lenterra in 2003 to commercialize opto-mechanical analytical technologies that he pioneered. Over his scientific career, Valery has worked in areas of optical spectroscopy, plasma aerodynamics, and micro-optical sensing. Valery received his PhD in physics from Saint-Petersburg State University in 1985.

Lenterra
Newark, NJ
973-623-0755
www.lenterra.com