White Paper 7

DFF Sensor Differentiates Powders of Different Particle Size

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Identification of Particle Size in Powders

When placed into a solvent-free powder flow, DFF sensor produces a time-varying force signal that generally can be described as a superposition of a continuous force due to a fine powder component of the flow, and a series of pulses, which represent separate impacts of granules (see also White Papers 4 and 5).

This note compares DFF signals for powders of same material density but containing particles of different size.

Experimental

Material

The test powder was produced by granulating a pharmaceutical placebo formulation consisting of 37% anhydrous lactose, 1% croscarmellose sodium, and 3% hydropropyl cellulose (HPC) with 57% microcrystalline cellulose with 40% water, wetmassed for 23 minutes in a 4L Bohle high shear granulator. After letting it dry, the powder was sieved first using a 3.2 mm and then a 0.8 mm mesh. The resulted sample powders contained granules of the following size characteristics:

P0: raw mixture containing particles of all sizes

P1: fine powder-particles below 0.8 mm
P2: from 0.8 to 3.2 mm
P3: above 3.2 mm (6 mm observed)

Largest particle size powder (P3) is shown on the photograph.

Method

The samples were analyzed in a free-fall apparatus described in White Paper 5. The powder was loaded to a vertically held aluminum pipe and released to a horizontally positioned DFF probe in a variation of hopper discharge.

Results and Discussion

Several loads for each sample powder were analyzed. Typical measurements for each sample are shown in the figures on the next page. Force was measured at a rate of 500 Hz or every 0.002 s. Raw signals are compared to force pulse magnitude (FPM) dependencies calculated for FPM frequency of 79 Hz that is the characteristic frequency of free probe oscillation (observed, for example, on the latter stages on the force vs. time signal). This is a top frequency in FFT spectra obtained from the raw signals using Lenterra's post processing software. White Paper 3 describes how FPM is calculated.

The total action of the powder lasted approximately 0.25 seconds for all samples except P3 where granules interacted with the probe for more than 0.4 seconds indicating reduced flowability of the large size particle loads.

Force from fine powder (P1) does not show sizable pulses. It is a steady signal consistent with action of fine powder or liquid as discussed in White Papers 2 and 5. The P1 raw signal steadily increases from the start (at 9.58 seconds) to the end (at 9.89 seconds) of the powder interaction with the probe, because powder falling from the upper parts of the tube has greater velocity when striking the probe pin. Individual particle impacts are not resolved therefore FPM signal is low. Relatively large FPM values at the beginning and the end of the measurement are due to initial and final impact of the load.

In the medium (P2) and large (P3) sized powders there are particles that are larger than the probe pin diameter. Single impacts of such particles provide much stronger instantaneous forces.

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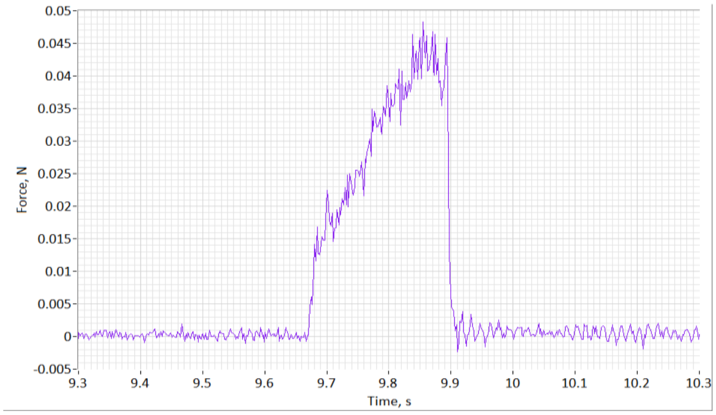
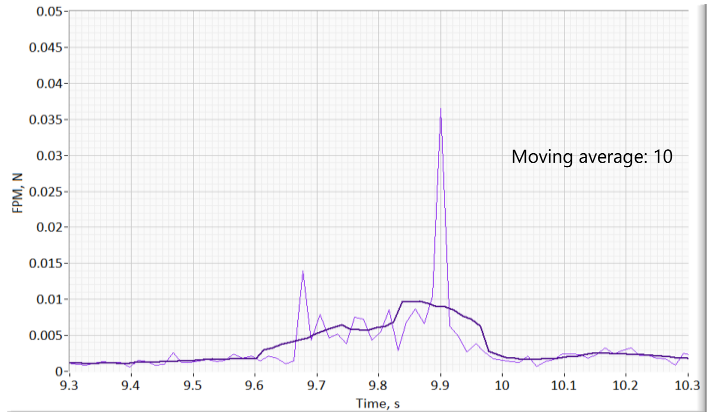
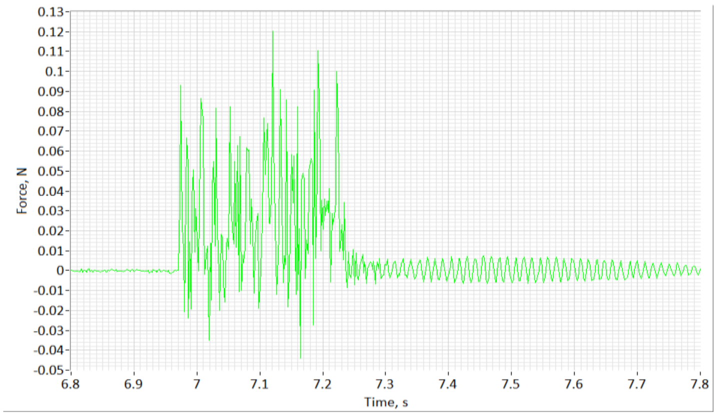
Force vs. time ***FPM vs. time***

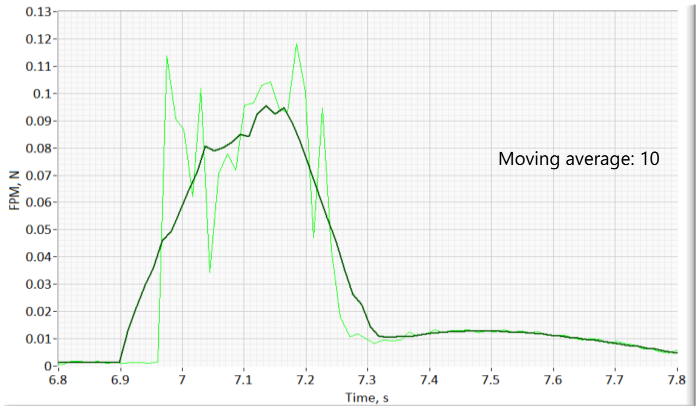
P1: Particle size below 0.8 mm



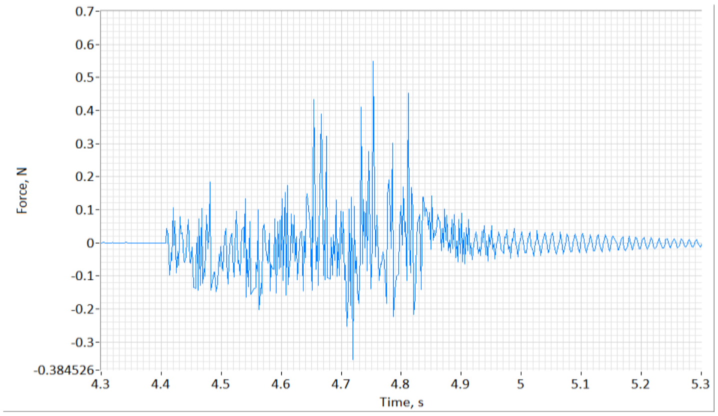


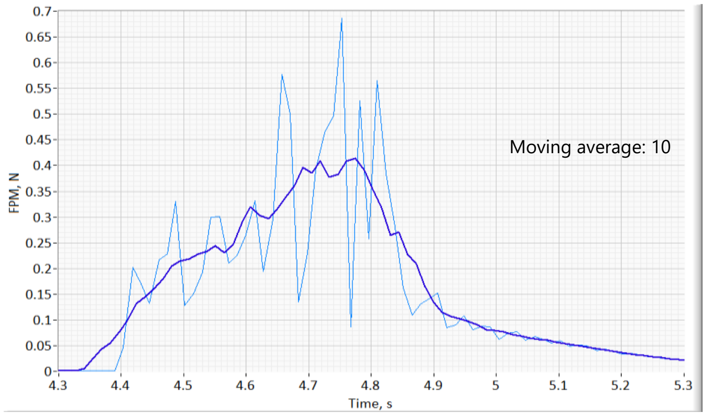
P2: Particle size between 0.8 and 3.2 mm



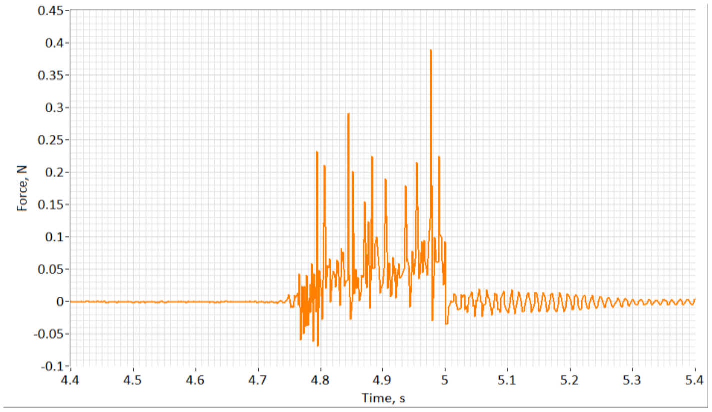


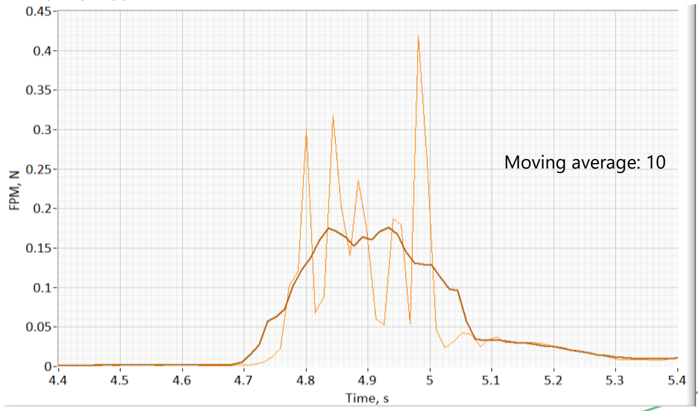
P3: Particle size above 3.2 mm





P0: Raw mix – all sizes





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After a single particle impact, the deflected probe starts oscillating around its equilibrium state and can show negative value for the force (see undisturbed oscillations after 2.3 sec for P2, 4.9 seconds for P3 or 5.0 seconds for P0). The oscillation continue until another particle interrupts it (dumping). Fine powder (or liquid) provides strong dumping since the impacts of its particles are very frequent. This is why large granule pulses in the raw mixture (P0), while showing magnitudes as large as in pure large particle sample (S3), do not rebound into negative values.

Presence of fine powder in sample P0 is recognized by a steadily rising continuous force (background) that is similar to that in sample P1. Note that this background is not present in the large granule size signal (P3).

Maximum value of FPM smoothened over 10 natural frequency oscillations (moving average 10) appears to be a reliable metric for differentiating particle size. These maxFPM(10) values are 0.01N, 0.09N and 0.41N for samples P1, P2, and P3, respectively. The raw mix demonstrated maxFPM(10) = 0.18N, an average value.

Conclusion

In a type of hopper discharge, a Drug Force Flow (DFF) sensor reliably separated loads of powders of three different particle size composition. For a fine powder with maximum particle size smaller that the DFF probe pin diameter, the DFF sensor measured a steadily rising continuous force, and the DFF signals for larger granule sizes consisted from multiple pulses each characterizing a single granule impact. The proposed metric, maximum value of force pulse magnitude smoothened for 10 periods of probe natural oscillation, maxFPM(10), reliably separated the three powder sizes tested. DFF sensor therefore demonstrated itself as a highly promising process analytical technology (PAT) tool for applications involving solvent-free powders.