APPLICATION NOTE 7

DFF Sensor Response to Powders with Different Particle Size

Valery Sheverev and Vadim Stepaniuk

Identification of Particle Size in a Powder

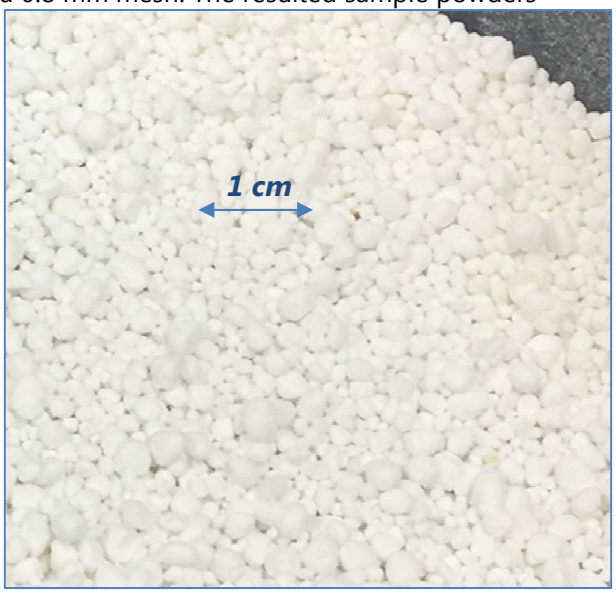
Being placed into a powder flow, DFF sensor produces a time-varying force signal that generally can be described as a superposition of a continuous background signal caused by a liquid and/or fine powder component of the flow, and a number of force pulses or peaks, which represent separate impacts of granules (see also Application Notes 04 and 05).

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: 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

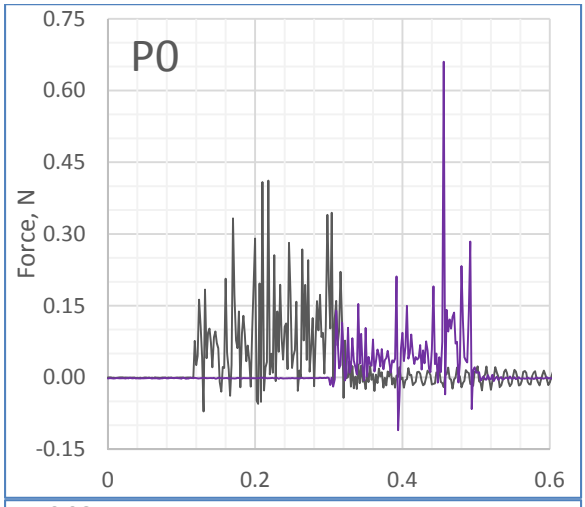
The samples were analyzed in an apparatus where powder was loaded to a vertically held aluminum pipe and released to a horizontally positioned DFF probe in a variation of hopper discharge (see Application Note 05 for details).

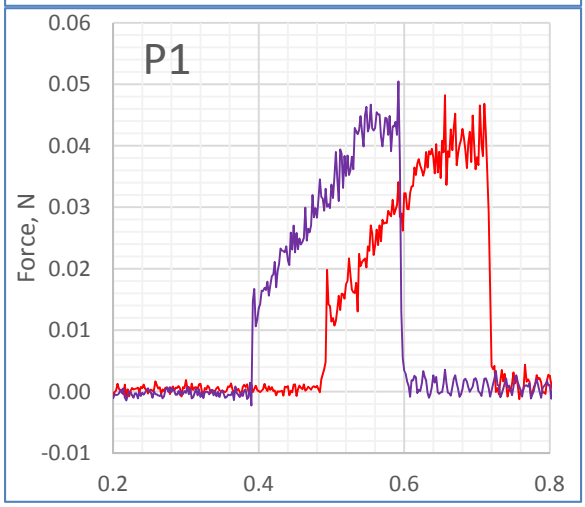
Results and Discussion

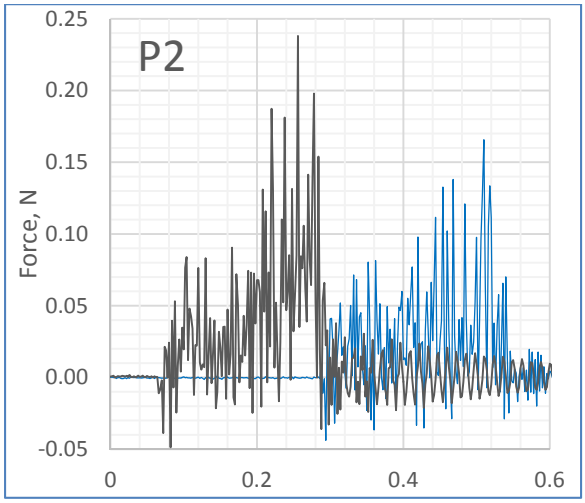
Raw Signal

Five consecutive runs for each sample powder were analyzed. DFF sensor raw signals for two characteristic runs from each set (except P3 where one run is shown) are compared on next page. Horizontal axis represents time in seconds. Force measurement rate was 500 Hz or every 0.002 s.

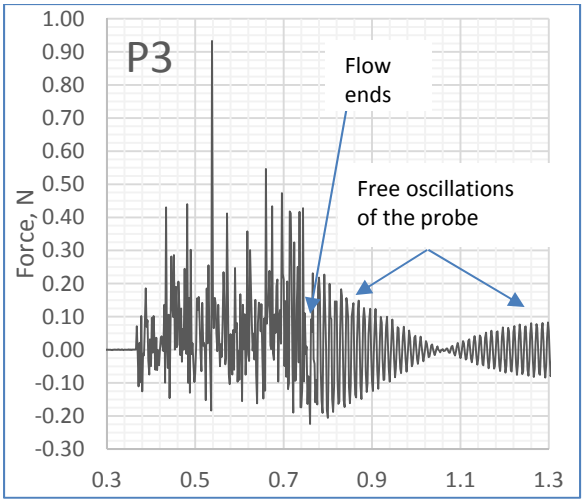
Fine powder (P1) represents the background signal that increases to the end of the run where the velocity of the powder is highest. The total action of the powder lasted approximately 0.25 seconds for all samples except P3 where granules interacted with the probe for almost 0.4 seconds indicating reduced flowability of the large size particles. Signals for P3 also characterized with a strong oscillation of the probe after the flow ended, which is not as well pronounced in the raw mixture containing similar size granules (P0). Fine powder within the mix damped the oscillation. Force pulse magnitudes (FPMs) in powder P2 were up to five





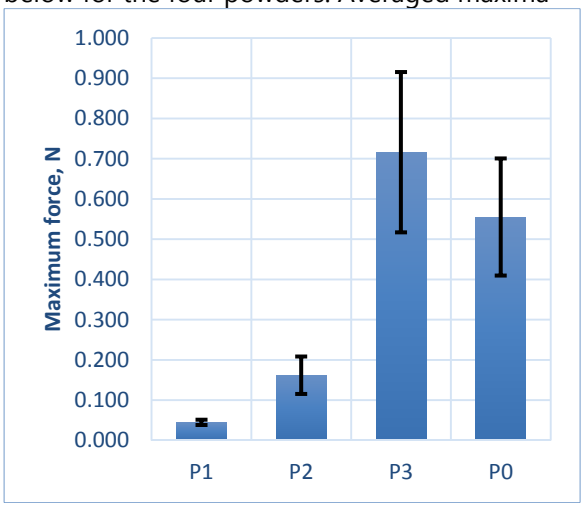


times greater those of the fine powder P1, and those observed for large granules (P3) were further almost four times greater. The raw mixture P0 contained all kind of magnitudes, with large peaks appearing infrequently in the signal.



Maximum FPM

The peak force observed in each run is tabulated below for the four powders. Averaged maxima



Max FPM, N

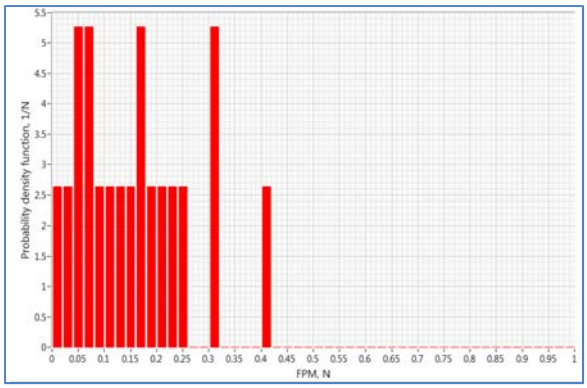
	Run 1	Run 2	Run 3	Run 4	Run 5	Mean	St. dev
P1	0.048	0.048	0.042	0.034	0.050	0.045	0.007
P2	0.120	0.155	0.166	0.238	0.129	0.162	0.047
P3	0.548	0.864	0.763	0.933	0.472	0.716	0.199
P0	0.390	0.607	0.707	0.412	0.660	0.555	0.146

are plotted below. The error bars represent standard deviation.

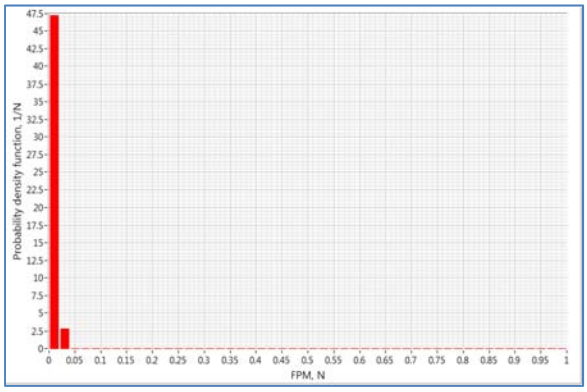
Histogram Statistics

Histograms of force pulse magnitudes (FPM, see Application Note 03) calculated for FPM frequency of 78 Hz, which is the natural mechanical frequency of the DFF probe used in the tests, are shown for one representative run from each of the four powders.

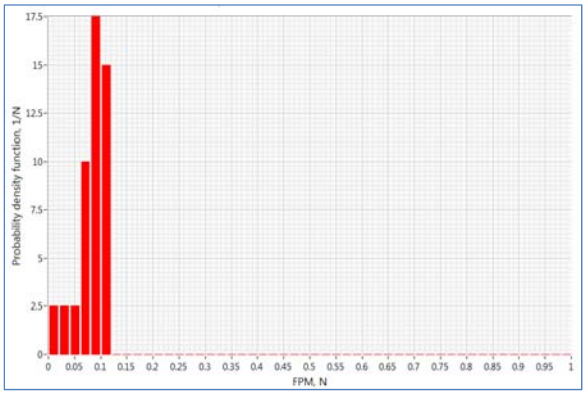
P0, bin size 0.02N



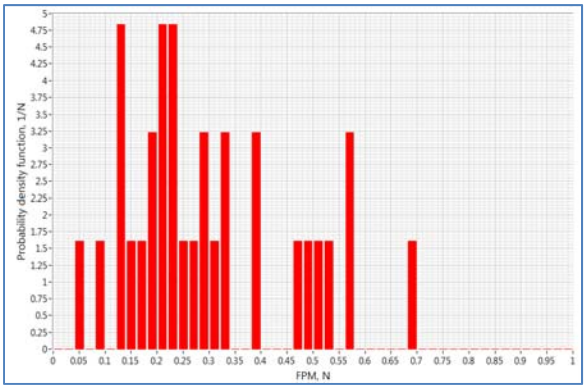
P1, bin size 0.02N



P2, bin size 0.02N



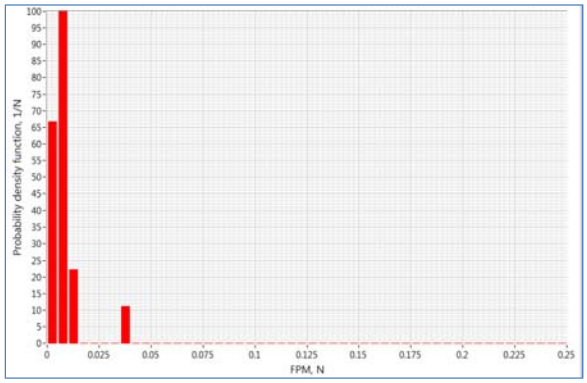
P3, bin size 0.02N



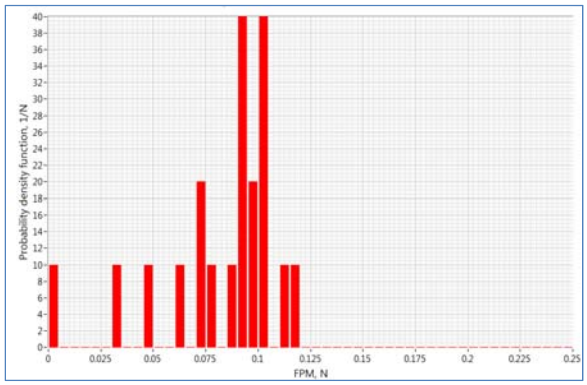
The difference between the powders is the increasing number of higher magnitude peaks for samples with larger particle size. For the raw mix (P0) the distribution is most uniform reflecting the fact that the mix includes particles of all sizes.

Diferentiation between samples P1 and P2 is better seen on histograms with a smaller bin size of 0.005 N, that are compared below:

P1, bin size 0.005N



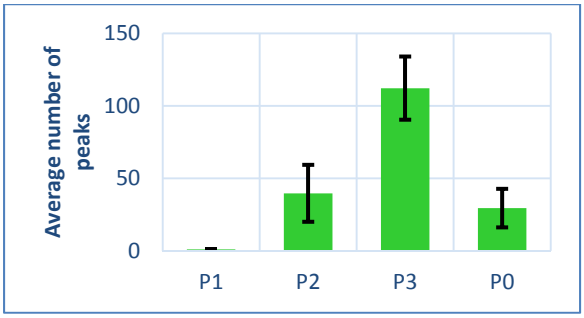
P2, bin size 0.005N



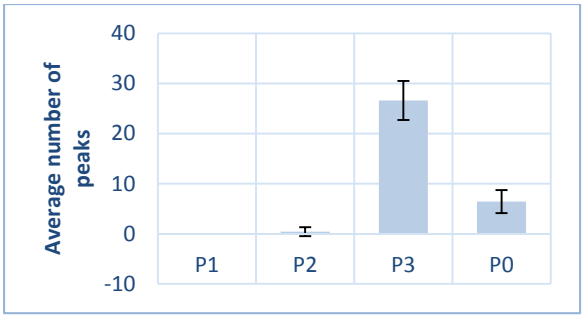
Threshold Analysis

A single parameter that could be used for differentiation of powders of different particle size is the number of FPMs above a certain threshold. Tables below compares the number of FPMs above 0.02 N and 0.2 N, respectively for each of the runs in the test. Averaged values for each sample are plotted together with error bars representing the standard deviation among the five runs.

Number of peaks above 0.02N						Mean	St. dev
	Run 1	Run 2	Run 3	Run 4	Run 5		
P1	1	1	1	1	1	1	0
P2	20	66	23	36	53	39.6	19.7
P3	91	94	119	145	112	112.2	21.8
P0	34	16	45	37	15	29.4	13.3



Number of peaks above 0.2N						Mean	St. dev
	Run 1	Run 2	Run 3	Run 4	Run 5		
P1	0	0	0	0	0	0	0
P2	0	0	0	2	0	0.4	0.9
P3	22	23	31	29	28	26.6	3.9
P0	5	7	6	10	4	6.4	2.3



Conclusion

Installed below a pipe filled with a powder, in a type of hopper discharge, a Drug Force Flow (DFF) sensor by Lenterra Inc reliably separated powders of three different particle size composition, one containing particles below 0.8 mm size, another with sizes between 0.8 and 3.2 mm, and the third from 3.2 to approximately 6 mm. The DFF signals from the three powders were characterized by a number of force pulses, which magnitude and amount varied significantly from one powder to the other, both parameters increasing with particle size as seen from the peak magnitude histograms. Either a maximum pulse magnitude or a number of peaks above a threshold, for thresholds are suggested as a convenient single parameter characterizing segregation of powders for particle size.