White Paper 15

Characterizing dry powders: common sugars

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A pair of MFPM and PCF values were measured for a number of commercially available sugar powders using a DFF sensor station. The DFF Test Station is a 2-liter volume bottom-driven mixer with a DFF probe installed vertically in such a way that probe was positioned 8 mm above the agitator upper blade as shown in Figure 1.

In every test, the mixer was loaded with 1.6 liters of test powder so that the 4-cm probe was just covered with powder. The test protocol involved running the mixer for 60 seconds and taking measurements at a rate of 500 per second. The agitator rotational speed was kept at 600 RPM. MFPM, WFPM and PCF values (see [1,2] and White Paper 13 for definition of the metrics) were calculated for the array value of 200, and the MFPM and PCF values at a time instant of 30 seconds were collected in each test. Three consecutive tests were taken for the same load.

Figure 2 shows an exemplary raw force signal collected in a first test with brown sugar. The "forest" of force peaks (upper plot) consists of elementary pulses (lower plot) formed by powder driven by the agitator blades and interacting with the DFF probe. Figure 3 displays the MFPM and PCF evolutions in the vicinity of the 40th second of the test calculated using the raw data of Figure 2. The collected values of the MFPM and PCF for each of the three tests are shown in Figure 3 with respective arrows.

Table 1 and Figure 4 compare the MFPM and PCF results for seven commercially available sugar powders. Error bars represent standard deviation over three measurements for each powder.

Table 1:

Material	MFPM, N	PCF
Brown Sugar	0.415	8.7
Augason Farms Sugar	0.220	21.8
Domino Sugar	0.170	22.9
Coconut Palm Sugar	0.156	13.3
Caster Sugar	0.135	26.4
Confectioners Sugar	0.033	6.0
Corn Starch	0.018	32.8

Brown sugar, characterized by largest granules, displayed MFPM values that are about two times greater than those for the four granulated sugars and about an order of magnitude greater than those for the powder sugar (Confectioners) and corn starch. The MFPM values generally correlate with the average granule size of the

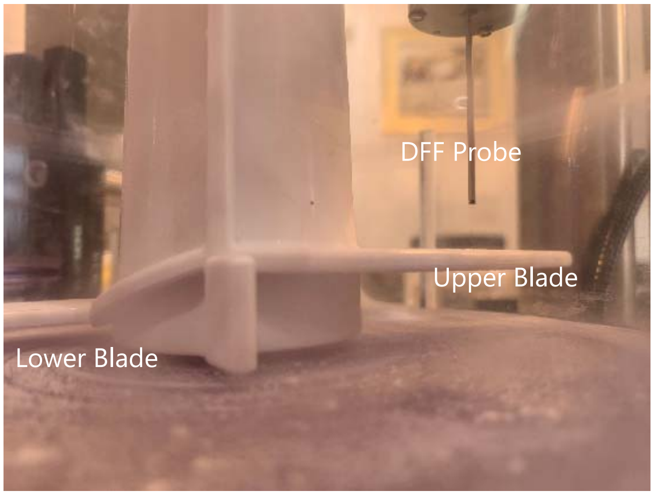

Figure 1. DFF probe installed inside the test mixer.

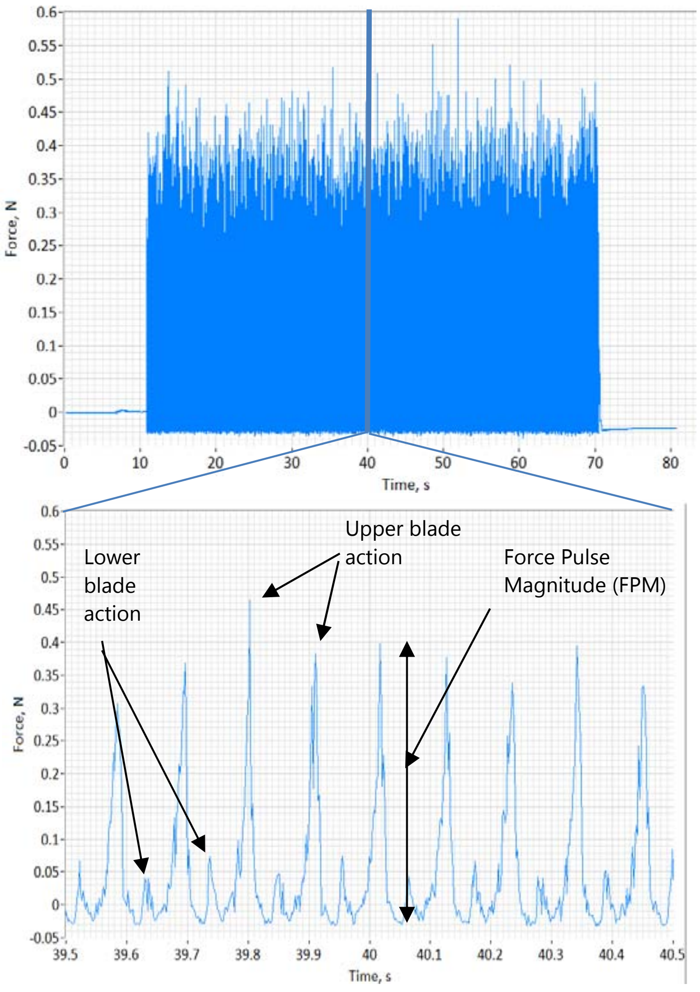

Figure 2. The total signal (upper plot, 40000 points) and its one-second detail (lower plot, 500 points) obtain in a DFF test station for a load of commercial brown sugar.

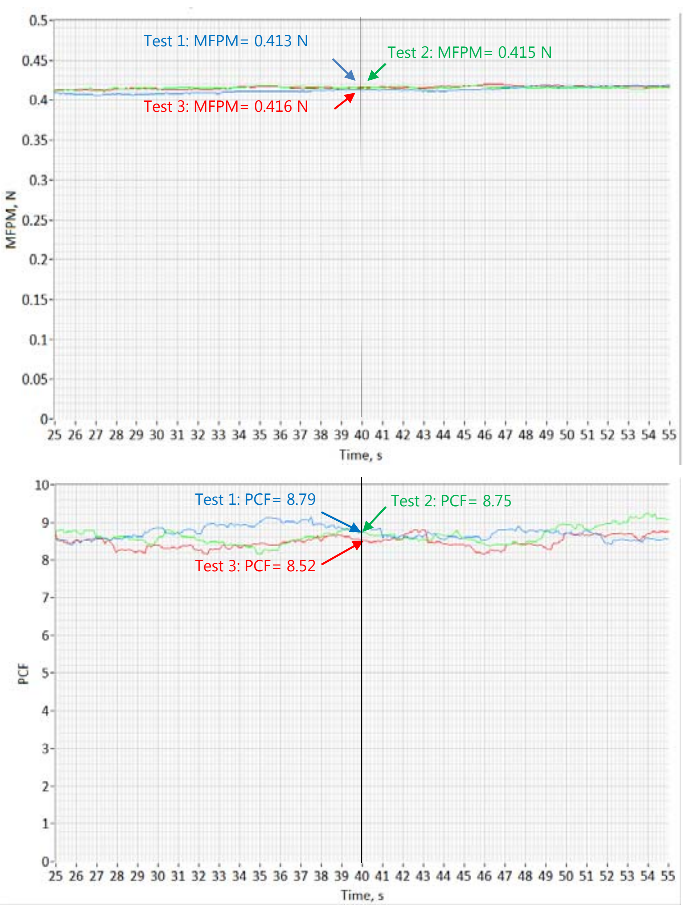

Figure 3. MFPM and PCF dependencies for three tests of brown sugar. MFPM and PCF were calculated for the FPM array size of 200. The three values for both MFPM and PCF at 40 seconds were collected. The procedure was repeated for other sugars, see results in Figure 4.

powder.

The powder consistency factors for Confectioners sugar, Coconut Palm sugar, and Brown sugar are found to be significantly smaller than that for other powders. A cohesive and tacky material forms agglomerates that randomly strike the probe resulting in FPM values that significantly exceed the average FPM. Such strong infrequent pulses do not noticeably affect the average value (MFPM) but significantly widen the FPM distribution and thus decrease PCF. Indeed, these three sugars were found being sticky on touch.

Conclusion

The DFF Test Station reliably separated the tested powder, with very good repeatability. The high sensitivity and repeatability of DFF powder characteristics (MFPM and PCF) stem from 1) high sensitivity of force measurement by the DFF probe that leads to precise measurements of force pulses provided by the blade, and 2) high measurement rate of 500 samples per second that allows for averaging a large number of FPMs over short period of time (200 in 20 seconds, in these tests).

fingerprint (MFPM + PCF) that characterises both particle size and density, as well as cohesiveness of the powder.

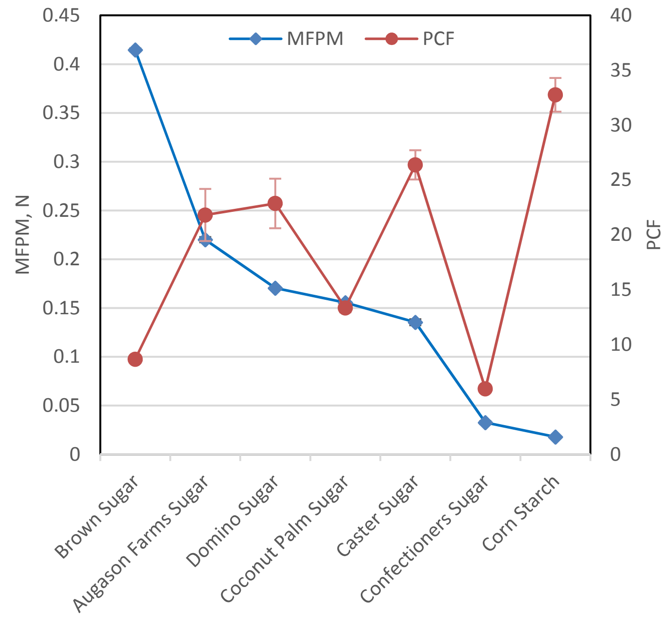

Figure 4. Comparison of a pair of MFPM and PCF values measured for seven commercially available sugar powders. Each point is an average of three values collected for each powder, as illustrated in Figure 3. Error bars represent standard deviation over the three measurements.

This information may help differentiate powders from different manufacturers and provide a metric for various processes that may help to reduce product variability (such as in tablets or various food products).

References

1. Valery Sheverev, Vadim Stepaniuk and Ajit S. Narang, *Principles of Drag Force Flow Sensor*, in “Handbook of Pharmaceutical Wet Granulation”, Ed. Ajit Narang and Sherif Badawy, Academic Press 2019, Ch. 15.
2. Ajit S. Narang et. al., *Real-Time Assessment of Granule Densification and Application to Scale-up*, Handbook of Pharmaceutical Wet Granulation, Ed. Ajit Narang and Sherif Badawy, Academic Press 2019, Chapter 19.