White Paper 16

Comparative Study of Sugar Powder Consistency using an FT4 Powder Rheometer and Drag Force Flow (DFF) Sensor

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The rheological properties of seven common sugar powders were characterized using an FT4 Powder Rheometer® and a Lenterra Drag Force Flow (DFF) sensor. Data generated by both the FT4 Powder Rheometer and DFF sensor reflect mechanical resistance of the powders to forced flow. During dynamic flow FT4 analysis, a precise volume of powder is subjected to the flow pattern of a rotating blade that moves downward through the powder bed. Measurements of the torque and force applied to the blade are combined to calculate Basic Flowability Energy (BFE), quantifying the powder's resistance to the blade motion. [1,2,3]

The recently developed DFF sensing technology provides instantaneous readings of the force exerted by particulate flow to a narrow pin immersed within process equipment. DFF sensor quantifies the drag force, which is a function of material properties such as particle size, morphology and adhesion [4,5]. This in-line process monitoring instrument has shown to be a valuable Process Analytical Technology (PAT) tool for High Shear Wet Granulation (HSWG) [6,7]. For this study, an off-line DFF Test Station was assembled, where a probe was installed into a laboratory scale mixer with a bottom-mounted impeller.

In-line DFF sensor measurements have previously been compared to FT4 BFE data during HSWG process cycles [7]. The study confirmed that both the in-line DFF sensor and at-line FT4 similarly responded to changes in wet mass densification during the granulation cycle for the majority of blends.

The work reported here is focused on improving the comparison protocol by taking FT-4 and DFF measurements synchronously.

Equipment and test protocols

A 50 mm diameter test vessel containing 160 ml of powder was used for the FT4 analysis. Each sample was subjected to conditioning, where a precision blade was used to create low stress, homogeneous particle packing. Prior to testing, the vessel was also split to produce a consistent volume of materials for analysis. BFE measurements were conducted using an aggressive compaction mode, measuring the resistance to the motion of the rotating blade as powder was forced down in front of it.

The DFF sensor outputs a Force Pulse Magnitude (FPM)

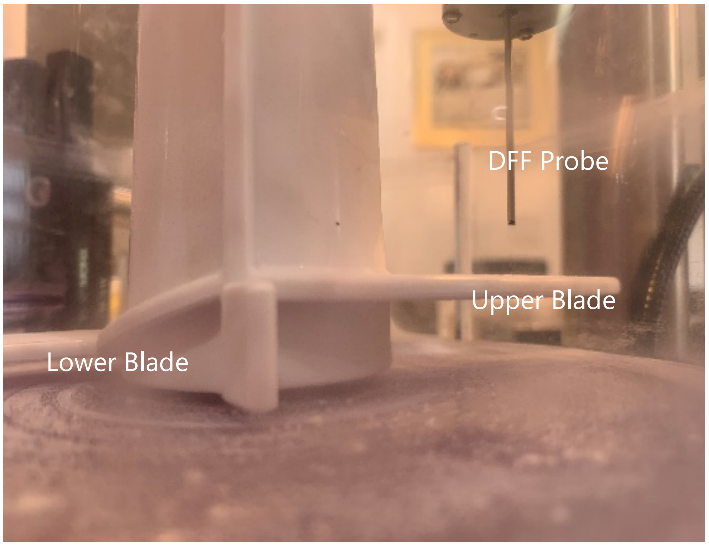

Figure 1. DFF probe inside the DFF Test Station mixer.

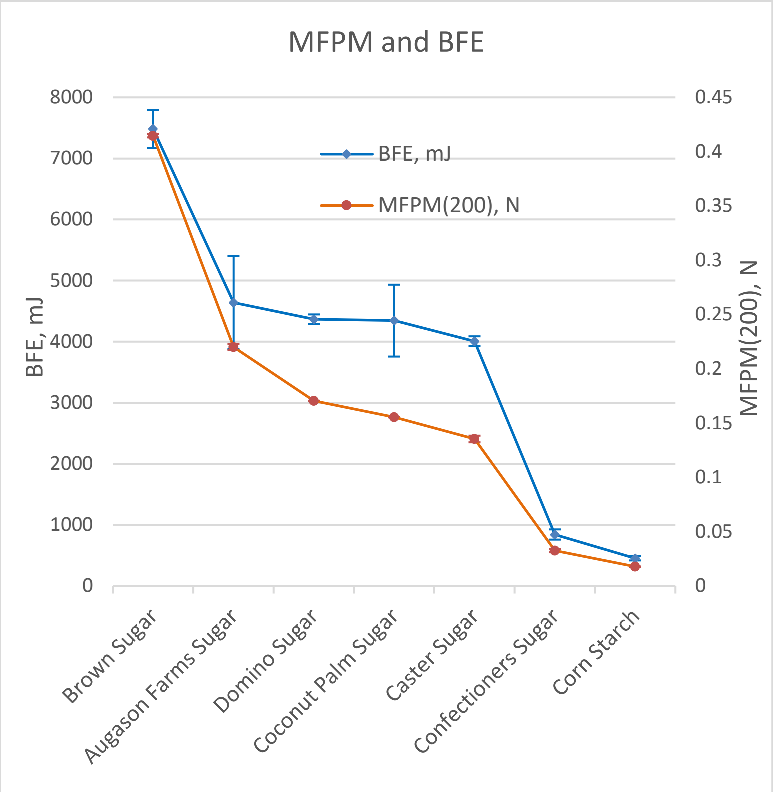

Figure 2. Comparison of BFE measured by FT4 and MFPM measured with the DFF test station. MFPM represents FPM averaged over 200 consecutive blades. See Appendix for more details of FT4 and DFF measurements. Error bars represent standard deviations over three tests.

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which characterizes the action of each passage of the impeller in the vicinity of the probe tip. Based on an array of FPMs, three characteristic metrics are calculated: a moving average of the mean FPM (MFPM), the width of the FPM distribution in the array (WFPM), and a Powder Consistency Factor (PCF) which is the ratio of MFPM and WFPM for a given FPM array. MFPM is a measure of bulk powder resistance, whereas PCF characterizes the uniformity of the powder, related to adhesion or cohesivity of the material. For more information on FPM, MFPM, WFPM and PCF please see reference [8].

In the DFF Test Station, the sensor was installed vertically so that the probe tip was 8 mm above the impeller, within the 2 L mixer as shown in figure 1. For each test, the DFF Test Station mixer was loaded with 1.6 L of powder so that the probe was fully immersed with material. DFF test protocol included running the mixer for 60 seconds and taking flow force measurements at a rate of 500 per second. The agitator rotational speed was approximately 600 RPM. Consistent with the FT4 measurements, the DFF tests were repeated in triplicate and the mean value used for comparison. More detail on DFF measurements in the test station can be found in White Paper 15 [9]

The value of MFPM after 30 seconds of mixing was compared to the BFE values obtained for the same powder during FT4 analysis.

Results and Discussion

Table 1 and Figure 2 compare the BFE and FPM results for the powders. Error bars represent standard deviations over three tests taken for each sugar.

Table 1.

	FT4	DFF	
Material	**BFE, mJ**	**MFPM, N**	**PCF**
Brown Sugar	7484	0.415	8.7
Augason Farms Sugar	4641	0.220	21.8
Domino Sugar	4369	0.170	22.9
Coconut Palm Sugar	4344	0.156	13.3
Caster Sugar	4007	0.135	26.4
Confectioners Sugar	842	0.033	6.0
Corn Starch	452	0.018	32.8

Both devices demonstrated highly comparable trends in BFE (FT4) and MFPM (DFF test station) for the seven different powders tested. Both methodologies categorized the powders into three distinct groups: brown sugar, four granulated sugars (Augason Farm, Domino, Coconut Palm and Caster Sugar) and fine powders (Confectioners' Sugar and Corn Starch).

Brown sugar, characterized by its large tacky granules,

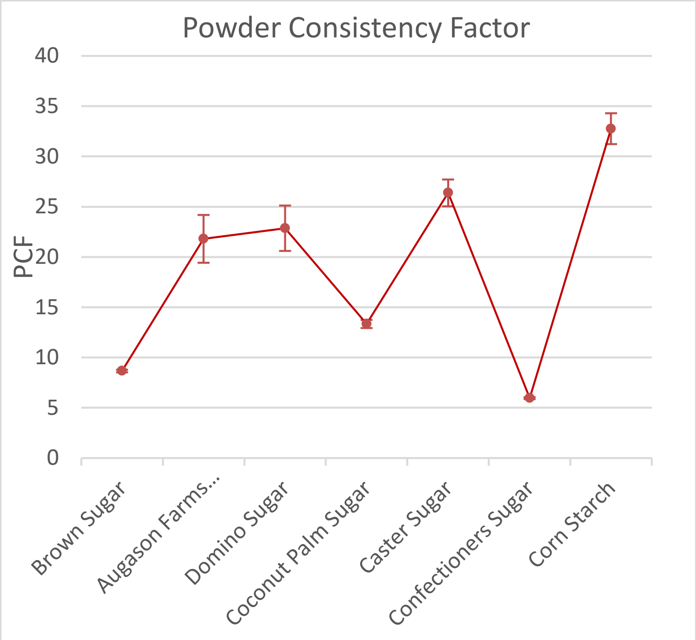

Figure 3. Powder Consistency Factor measured with the DFF Test Station. Error bars represent standard deviations over three tests. Brown, Coconut Palm and Confectioners sugar powders display lowest values of PCF which reflects their higher cohesiveness.

displayed significantly higher BFE and MFPM values, approximately two times greater than the values generated by the four granulated sugars and around an order of magnitude greater than the fine powders. Corn starch was found to demonstrate the lowest value of BFE and MFPM, with Confectioners Powdered sugar coming in close second place for both techniques.

Differences between MFPMs measured for four granulated sugars exceeded the standard deviations by at least a factor of five, highlighting the excellent sensitivity of the DFF technique. The high sensitivity and repeatability of DFF measurements stem from both the high sensitivity of force measurement by the DFF probe, and high measurement rate of 500 samples per second.

The DFF sensor also reported the Powder Consistency Factor (PCF), which is a measure of flow uniformity for each sugar (figure 3, see also [9]). PCF factors for Confectioners' Powder sugar, Coconut Palm sugar, and brown sugar were found to be significantly smaller than that of the other sugars studied. This is consistent with the high cohesivity observed for these powders.

The two finer powders, Confectioners' Sugar and Corn Starch, displayed MFPM values that differed by a factor of two, and the PCF values that differed significantly by a factor of six. This confirms that the MFPM and PCF parameters reflect different properties of the powder.

Conclusions

The DFF test station and FT4 Powder Rheometer® were used to concurrently measure characteristics of commercially available sugar powders. This study has demonstrated that both the DFF sensor and FT4 Powder Rheometer can identify differences between a variety of different sugar powders. The comparable trends observed between the BFE and DFF data demonstrate how both of these techniques can be used to quantify the material's resistance to forced, dynamic flow.

References

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[5] http://lenterra.com/wp-content/uploads/2018/05/WP-_01_DFF-Principles.pdf

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