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Analysis of the Periodic Motion in a Powder Rheometer and Development of a New Flowability Testing Method[†]

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Abstract

This study focuses on the periodic motion observed in the FT4 Powder Rheometer and aims to develop a new testing method to evaluate powder flowability. This new method is based on the autocorrelation analysis of the torque measurements. The test results of powders belonging to different Geldart's groups show that the torque measurements are periodic and that the cycle time (period) depends on the flowability. When the powder cohesiveness increases, the oscillations' amplitude and the cycle time increase. Conversely, a free-flowing powder exhibits a liquid-like behavior and shows almost no periodic motion.

Keywords: powder rheometer, flowability, periodic motion, autocorrelation

1. Introduction

Powders or bulk solids are widely used in the industries. A good powder flowability is of crucial importance since poor powder flow can lead to many problems during manufacturing such as inaccurate dosing and off-specification products (Valverde J.M., 2012). Especially during a number of processes such as filling small bags or tablet press dies, the powder materials are under very low stress conditions and their flow behavior is more difficult to predict (Zafar et al., 2015).

To replicate and characterize the powder flow at low stress conditions, a number of characterization techniques have been developed over the last 50 years (Jenike A.W., 1980; Prescott J.J. and Barnum R.A., 2000; Schwedes J., 2003; Zafar et al., 2015).

The shear cell test is most commonly used for the characterization of powder flowability. It gives the relationship between the shear stress (which leads to flow of the material) and the normal stress (Bek et al., 2016). The yield locus in the low stress region and the tensile strength are obtained indirectly by extrapolating to zero of the measured yield locus. However, the yield locus tends to curve downwards at low stresses for cohesive powders so that the results can be overestimated (Vasilenko et al., 2013).

Other techniques that allow powder flow characteriza-

tion under low stress conditions are reported in the literature. The Sevilla Powder Tester (SPT) and the Raining Bed Method (RBM) are two similar test methods that can directly measure the tensile yield stress at low stress levels. Both methods use a controlled gas flow to introduce small stresses in the powder bed. The tensile yield stress is then calculated by measuring the pressure drop across the powder bed at failure (Formisani et al., 2002; Valverde et al., 2000).

Another recently developed test method, the ball indentation method (BIM), measures the resistance to plastic deformation of powder materials at very low stress to assess the flowability (Hassanpour and Ghadiri, 2007). However, this method was originally designed for continuum solids and the extension to the testing of cohesive powder beds has only recently been analyzed (Pasha et al., 2013).

Zafar et al. (2015) proposed a comparison of these four techniques (shear cell, SPT, RBM and BIM), and showed that:

- The powder flow characterization under low stress conditions is greatly influenced by the stress history. Consequently, the initialization procedure has to be well controlled in order to obtain reproducible data;
- Since the distribution of stresses in granular materials is highly heterogeneous, it is important to ensure the failure plane occurs at the proper location and in a reproducible way.

Quintanilla et al. (2006) studied the behavior of a series of granular materials of different cohesiveness in a half-filled and slowly rotating horizontal drum. The authors used a CCD camera and digital image processing to de-

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tect and characterize the avalanches. By statistical analysis, they studied the periodicity of the signals as well as the probability distributions of the avalanche size and the maximum angle of stability. The results showed that the system cohesiveness was correlated to these probability distributions: the larger the cohesion, the larger the average size and maximum angle of stability of the avalanches.

More in-depth studies and new testing methods still need to be developed to better characterize powder flow properties under low stress conditions. This work aims to develop a new dynamic testing method to evaluate powder flowability under unconfined flow conditions. This new method is based on the autocorrelation analysis of the torque measurements obtained with the FT4 Powder Rheometer.

2. Materials

Six powders belonging to Geldart's groups A, B and C were used in this study. The properties of these six powders are summarized in **Table 1**. The particle size distributions were measured using a laser diffraction size analyzer (Mastersizer 2000, Malvern Instruments) and

the particle density by a helium pycnometer Accupyc 1330 (Micromeritics). In addition, a liquid honey was used in this study as a reference material to simulate an "ideal" flow behavior. As it will be shown later in this paper, the liquid honey is viscous enough to obtain significant torque measurements. At the same time, these torque measurements are close to those corresponding to a "perfectly flowing" granular material. As a consequence, it will be considered as a "reference material" and will be used to simulate an "ideal" flow behavior.

3. Periodic motion during dynamic measurements

3.1 Standard dynamic test on the FT4 powder rheometer

Tests on the FT4 powder rheometer are based on a sophisticated twisted blade which passes through the powder bed along a predetermined helical path (**Fig. 1**). When the blade moves clockwise (conditioning cycle), the powder is lifted up and conditioned to obtain a uniform and reproducible state. When the blade moves anticlockwise (test cycle), it creates a compaction zone which is a local-

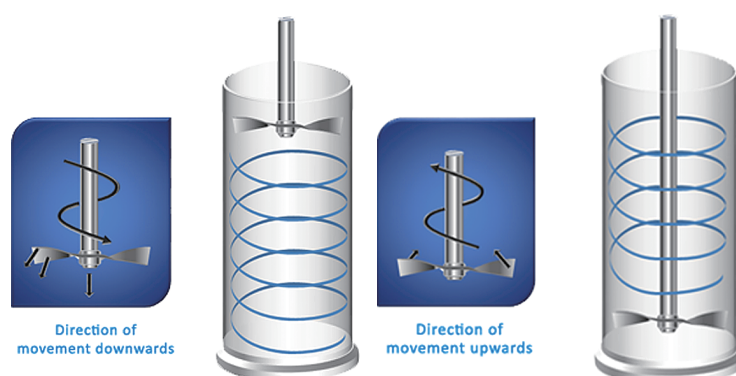


Fig. 1 Test cycle (left) and conditioning cycle (right) with the FT4 Powder Rheometer.

Table 1 Physical properties of the powders.

powder	$d[3, 2]$ (μm)	$d[4, 3]$ (μm)	$d[10]$ (μm)	$d[50]$ (μm)	$d[90]$ (μm)	span	ρ (kg/m^3)	shape	Geldart's classification
CaCO_3 (a)	3.4 ± 0.5	11.7 ± 1.7	1.2 ± 0.2	8.6 ± 1.4	26.1 ± 3.9	2.9	2750	angular	C
CaCO_3 (b)	4.0 ± 0.3	15.9 ± 1.4	1.5 ± 0.2	12.3 ± 1.1	35.2 ± 2.9	2.8	2750	angular	C
CaCO_3 (c)	8.4 ± 0.2	51.5 ± 1.2	4.9 ± 0.4	35.2 ± 0.3	104.6 ± 2.1	2.8	2750	angular	C
ZrO_2	23.7 ± 0.5	60.1 ± 0.5	12.8 ± 0.2	55.1 ± 0.4	113.0 ± 1.2	1.7	5790	angular	A
SiC	79.1 ± 0.7	66.0 ± 3.6	43.4 ± 0.9	73.7 ± 0.5	122.7 ± 0.4	1.1	3056	angular	A
sand	342.2 ± 15.7	287.1 ± 8.1	186.3 ± 6.6	306.3 ± 11.4	517.6 ± 20.6	1.1	2546	angular	B

Values after "±" indicate the 95 % confidence intervals;
span = $(d_{90} - d_{10})/d_{50}$

ized high-stress region in front of the blade and forces the powder to flow (Leturia M. et al., 2014).

The FT4 standard “dynamic test” measures the evolution of the torque and force for a downward flow pattern in a conditioned controlled volume of powder. The energy required to induce this movement is then calculated (by integration of the force and torque measurements) to evaluate the flow properties of the powder (Conesa C. et al., 2004). This test is divided into two main parts.

The first part, called “stability test”, corresponds to seven identical complete cycles (conditioning + test cycles) at a constant blade tip speed of 100 mm/s. The objective is to detect any possible change in the powder flow properties after repeated tests (attrition, agglomeration, etc.). The stability test also allows a stable energy level to be achieved. The basic flow energy (BFE), as defined by Freeman Technology, corresponds to the stabilized flow energy. It represents the energy required to force a conditioned powder sample to flow during a downwards test:

$$BFE \text{ (mJ)} = \text{Flowenergy of test 7} \quad (1)$$

Usually, fine powders have relatively high interparticulate forces compared to gravitational forces. As a consequence, they behave in a cohesive way: Agglomerates are formed and a large amount of air is trapped inside the bulk powder (Freeman R., 2007). In this case, particles forced to flow at the blade face can be accommodated by the voids that exist between the agglomerates (high compressibility). The resulting flow or stress transmission zone is relatively localized. Thus the displaced volume of powder is small and the measured BFE value is particularly low. Powders made of larger particles often behave in a non-cohesive manner due to the minimal effect of cohesive forces compared to gravitational forces. Less air is trapped in the bulk powder resulting in a relatively lower compressibility. The flow zone is transmitted far ahead of the blade (Freeman R., 2007). In this case, the displaced volume of powder is large and the measured flow energy is high.

The second part, called “variable flow rate test”, corresponds to 4 complete cycles at decreasing blade tip speeds (100, 70, 40 and 10 mm/s). The objective is to evaluate the powder sensitivity to the flow rate. The flow rate index (FRI) corresponds to the ratio of the flow energy at 10 mm/s blade tip speed to the flow energy at 100 mm/s blade tip speed:

$$FRI = \frac{\text{Flowenergy of test 11 (10 mm/s)}}{\text{Flowenergy of test 8 (100 mm/s)}} \quad (2)$$

Many factors can affect the value of the FRI index, e.g.:

- Cohesive powders are more sensitive to the flow rate and present a high FRI value (Freeman R., 2007);
- Angular particles generally have a high FRI value.

Although the combination of the values of the BFE and

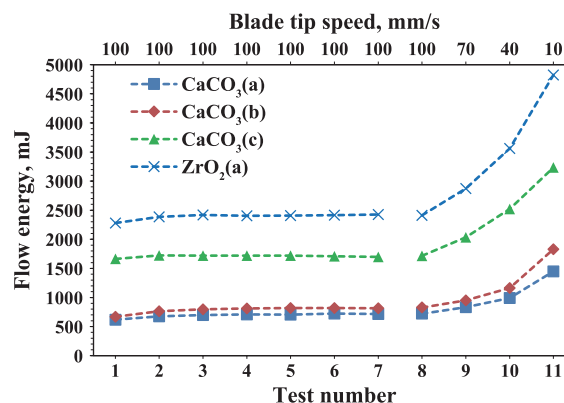


Fig. 2 Results of the FT4 standard dynamic test.

Table 2 Results of the FT4 standard dynamic test.

powder	BFE (mJ)	FRI
CaCO ₃ (a)	715.4	2.0
CaCO ₃ (b)	816.6	2.2
CaCO ₃ (c)	1696.2	1.9
ZrO ₂	2426.1	2.0

FRI helps to identify the powder flow properties, the standard dynamic test has many limitations for the classification of powder flowability.

Fig. 2 and Table 2 present the standard dynamic test results and the corresponding BFE and FRI values of four powder samples:

- CaCO₃ (a) has a slightly smaller BFE value than CaCO₃ (b), which generally means a lower flowability. On the other hand, CaCO₃ (a) has a smaller FRI value than CaCO₃ (b), which is generally associated with a better flowability;
- ZrO₂ has a much higher BFE value than CaCO₃ (c) and seems to have a better flowability. But ZrO₂ has a much higher bulk density, making it difficult to know whether the higher value of BFE is caused by the better flowability or the higher density. Meanwhile, ZrO₂ has a higher FRI value, which in contrast may indicate a poorer flowability.

In both cases, it is difficult to make a conclusion about which powder has better flowability. Therefore, the standard dynamic test on the FT4 cannot be used to compare two different powders and it can be difficult to distinguish between very similar materials.

3.2 Observation of a periodic motion

In order to optimize the FT4 standard procedures, the powder flow behavior during testing was closely observed (Fig. 3) and the resulting raw data were analyzed (normal force and torque measurements before integration for the

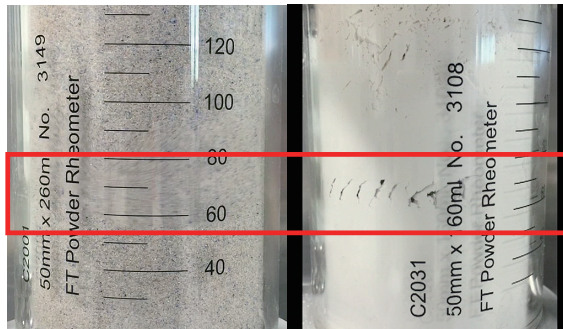


Fig. 3 Flow behavior of non-cohesive powders (sand, left) and cohesive powders (CaCO_3 (a), right).

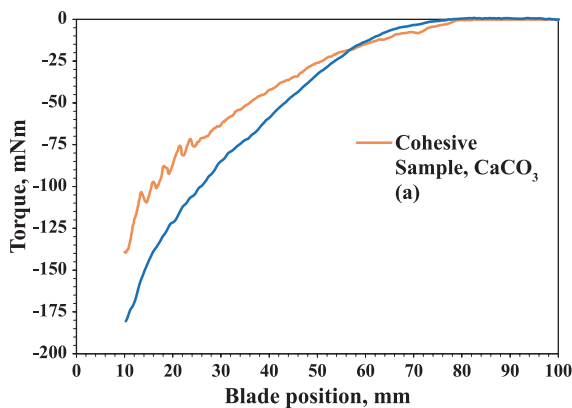


Fig. 4 Torque variation during a standard dynamic test (blade tip speed = 70 mm/s).

energy calculation). For cohesive powders, the blade leaves an empty space behind it when passing through the powder bed. This empty space grows in size as the blade moves forward and collapses when it reaches a maximum volume. It then grows in size and collapses again, following a cyclic pattern. It was also observed that this empty zone had a smaller maximum volume at a lower blade tip speed. For non-cohesive powders, the blade does not leave any significant empty space behind it and particles flow around the blade, almost like a liquid. The cyclic pattern is also reflected in the torque measurements where oscillations can be observed (**Fig. 4**). These oscillations are much more significant in a cohesive sample compared to a free-flowing powder.

A “verification test” was performed to reveal the causes of this cyclic pattern. In this test, only the surface of the powder bed was forced to flow so that the flow behavior could easily be observed. A 160-ml conditioned sample of CaCO_3 (a) was placed in the 50-mm ID cylinder vessel. The blade was moved just below the powder surface (70 mm from the bottom) and rotated for 60 seconds at 4 different blade tip speeds (100 mm/s, 70 mm/s, 40 mm/s and 10 mm/s). At the same time, a video camera recorded the powder motion around the blade. Since the blade

stayed at the same height, there was no significant normal force. Under these conditions, the total energy needed to move the blade through the powder is proportional to the torque acting on the blade. The torque oscillations were evaluated through an autocorrelation analysis, which is a mathematics tool commonly used in signal processing and time-series analysis to investigate repeating patterns (Dunn and Patrick F., 2005)

In statistics, the autocorrelation function for a continuous-time process, X_t , is the correlation of the process with itself at a later time. It represents the similarity between observations as a function of the time lag (Box G.E.P. et al., 1994):

$$ACF(\tau) = \frac{E[(X_t - \mu)(X_{t+\tau} - \mu)]}{\sigma^2} \quad (3)$$

where “E” is the expected value operator. For a stationary process, the mean μ and the variance σ^2 are time-independent and the autocorrelation depends only on the time lag τ . In other words, the correlation depends only on the time distance between the two chosen values but not on their position on the time axis. Particularly, the autocorrelation function of a periodic function has the same period as the original function, which is the key to investigating the repeating patterns. The values of the autocorrelation function (ACF) should be in the range $[-1, 1]$, with 1 indicating a perfect correlation (the signals exactly overlap when time is shifted by τ) and -1 indicating a perfect anti-correlation (Priestley M.B., 1982). For a discrete process with n observations such as the measured torque series in this test, an estimate of the autocorrelation may be obtained as (Chatfield C., 2004):

$$\widehat{ACF}(k) = \frac{1}{(n-k)\sigma^2} \sum_{t=1}^{n-k} (X_t - \mu)(X_{t+k} - \mu) \quad (4)$$

3.3 Analysis of the periodic motion

Fig. 5 presents the time evolution of the torque measurements and the corresponding autocorrelation functions (ACF) for the “verification test”. On the one hand, a series of oscillations in the torque measurements are observed and the autocorrelation function brings to light the existence of a cyclic pattern. At high blade tip speeds, the CaCO_3 (a) material has a period corresponding to one rotation of the blade. It should be noted that small peaks (small “events”) can be found at each half-rotation of the blade. As the blade tip speed decreases, it can be observed that:

- These small events become more significant and eventually reach the same value as the main peak. This means that the cycle time is halved;
- The maximum autocorrelation level decreases.

The test video was analyzed frame by frame. The im-

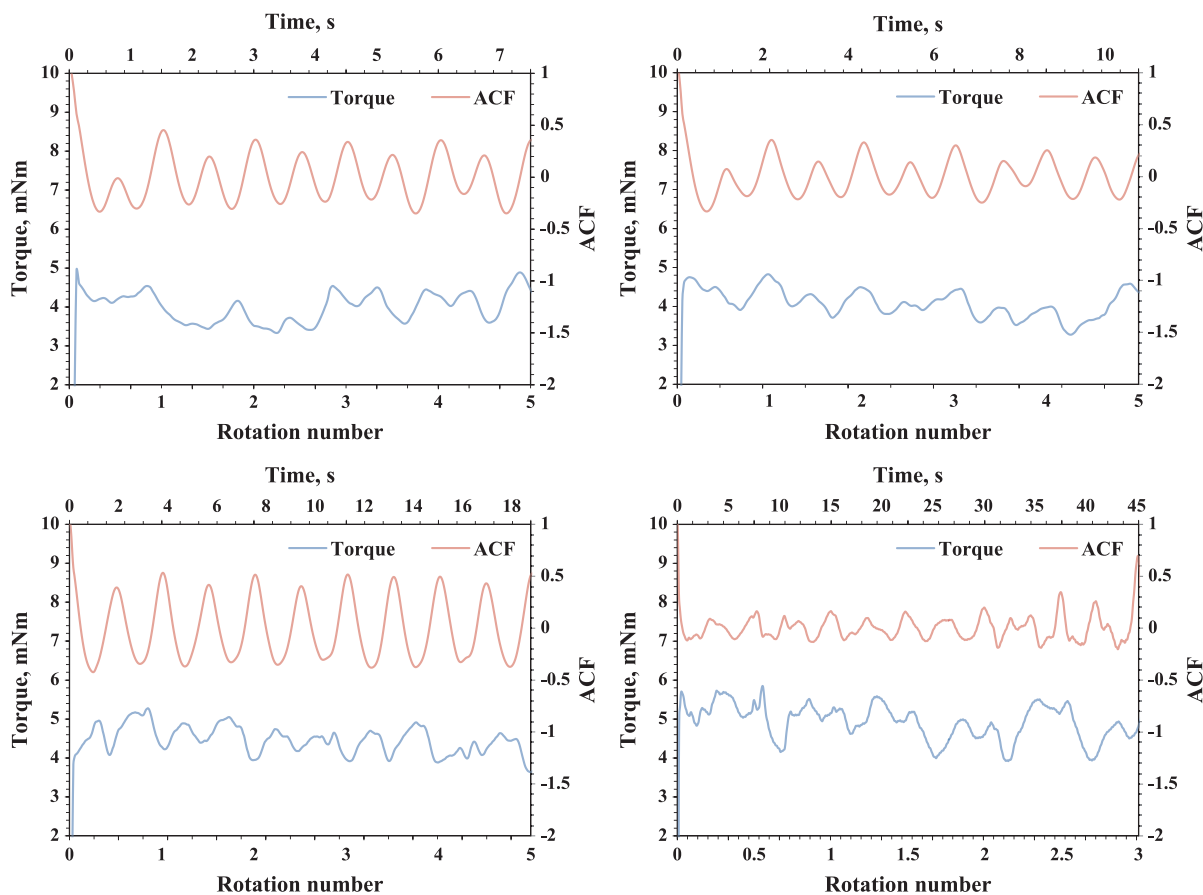


Fig. 5 Torque measurements and autocorrelation function (ACF) of CaCO_3 (a) for the verification test.



Fig. 6 Image of the verification test (the blade rotates in anti-clockwise direction).

ages clearly show (Fig. 6) that a “block” of powder moves in front of the blade while a large empty space is left behind it.

During the “verification test”, the “powder block” in front of the blade was observed in the form of several agglomerates (Fig. 7). Whenever some agglomerates fall off the blade (as the big one in the red frame in Fig. 7-A), a sudden drop is observed in the torque measurements

(Fig. 7-B). The torque goes up again when this agglomerate is picked up by the other side of the blade in Fig. 7-C, D. Since the agglomerates are not exactly of the same size, the oscillations do not have the same amplitude. In Fig. 7-E, F, G, H, torque oscillations can also be observed as several small agglomerates fall off the blade one after another.

Indeed, while the blade pushes the powder to flow, an unstable “compacted powder zone” builds up in front of each side of the blade, forming two “powder blocks” and two empty spaces behind them. Since these powder blocks are unstable, they may eventually collapse to fill the empty spaces. The fallen agglomerates stay in the empty space for a short time and are picked up by the other side of the blade. The second powder block then increases in volume and becomes less stable. It will finally collapse like the first powder block and this flow pattern is repeated continuously.

The flow speed has an important impact on this periodic motion:

- When the blade moves slowly, this process is quite smooth and the flow pattern has a period corresponding to a 1/2 rotation of the blade;
- When the blade moves fast, the cycle time of the flow pattern becomes one rotation of the blade.

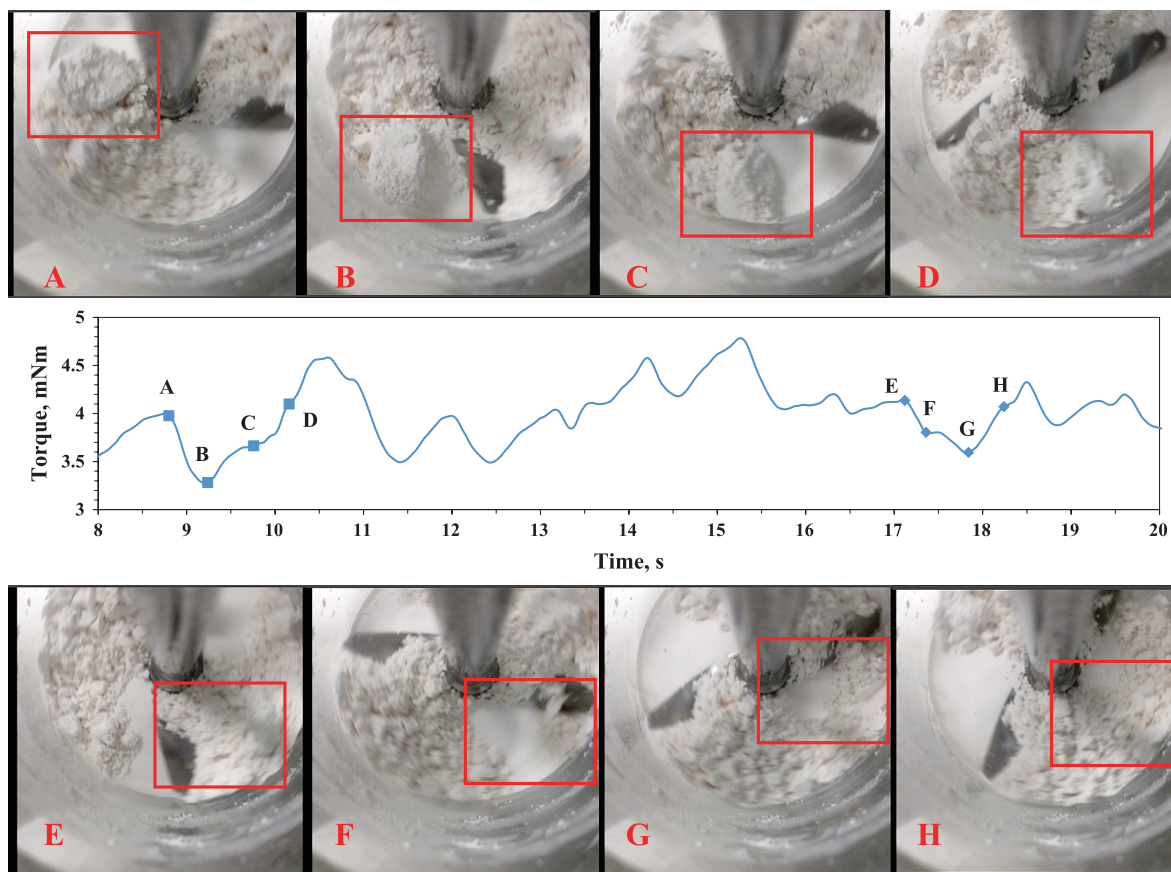


Fig. 7 Torque variation and agglomerates behavior (blade tip speed = 70 mm/s).

This behavior could be explained by the fact that the collapsing process is not immediate as a given powder has a certain response time to plastic flow deformation. As a consequence, there may be a critical flow speed above which the response time is longer than a 1/2 rotation, resulting in a cycle time of one complete rotation of the blade.

4. New powder rheometer testing method

A specific testing procedure was developed on the FT4 Powder Rheometer. First, a 160-ml conditioned sample was placed in the 50-mm ID cylinder vessel. Then the twisted blade was set to rotate in the powder bed at a height of 30 mm from the bottom with a blade tip speed of 100 mm/s for 200 seconds. The blade tip speed was then gradually decreased from 100 mm/s to 10 mm/s (by 10 mm/s steps for each speed reduction), and the rotation was repeated for 200 seconds at each step. A conditioning cycle was performed between each step.

The torque needed to induce flow was measured continuously. To ensure a stable powder flow (steady state), only the data measured during the last 50 seconds were analyzed for each step. The autocorrelation function was

then used to determine the cycle time and correlation level. By this means, the periodic motion was characterized and the powders could then be classified depending on their behavior.

4.1 Results obtained with the proposed testing method

The liquid honey was first used to simulate a regular flow behavior (reference material). Indeed, it was viscous enough to obtain significant torque measurements while behaving like an ideal material under the testing conditions. **Fig. 8 (top)** presents the variations of the required torque to force the liquid honey to flow at different flow rates. The measured torque is very stable (small oscillations) and the corresponding ACF values (**Fig. 8 bottom**) are always close to 0. This means that the torque needed to force a viscous liquid to flow at a given speed is a constant and no periodic motion can be detected with the FT4. From this point of view, the liquid honey can be regarded as an “ideal” material (in this flow rate range). On the other hand, it can also be noted that the average torque (**Fig. 8, center**) is proportional to the flow speed. This corresponds to the flow behavior of a Newtonian fluid.

The CaCO_3 (a), SiC and sand powders were first char-

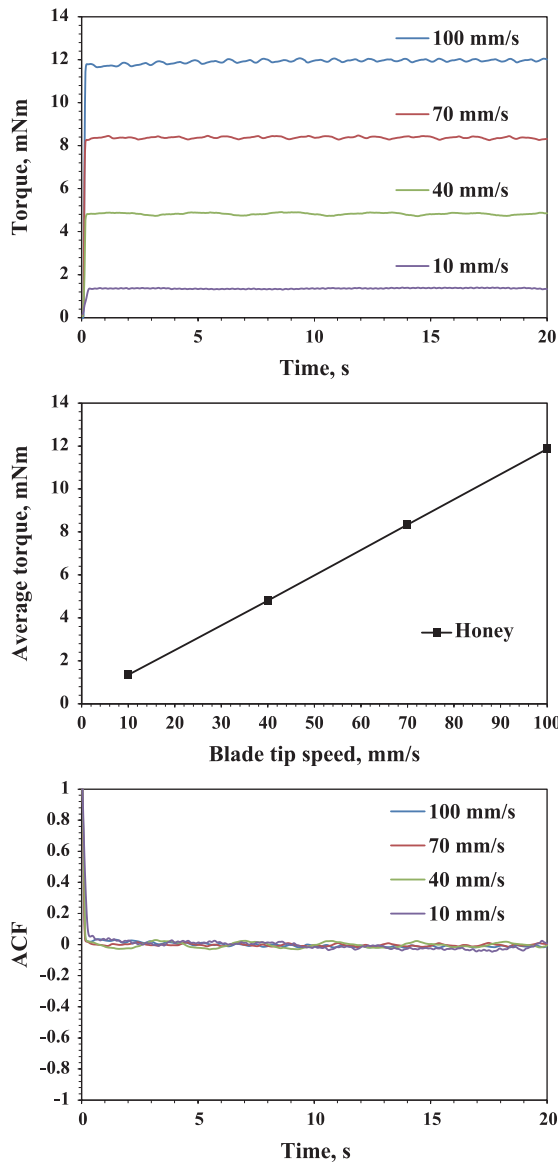


Fig. 8 Torque variation as a function of the blade tip speed for the liquid honey and autocorrelation analysis.

acterized by this method, since each of them belongs to a different Geldart’s group. Visual observations during the tests showed that:

- For the CaCO_3 (a) powder (group C), the rotating blade left a large empty space behind it by passing through the powder bed and only a small part of the powder moved in front of the blade;
- In the SiC powder bed (group A), the particles flowed around the blade, a small empty space was left behind and the flowing zone extended above and in front of the blade;
- For the sand powder (group B), the particles flowed freely, almost like a liquid. No empty space was left behind the blade and the flowing zone extended even further above the blade up until the surface of the powder bed.

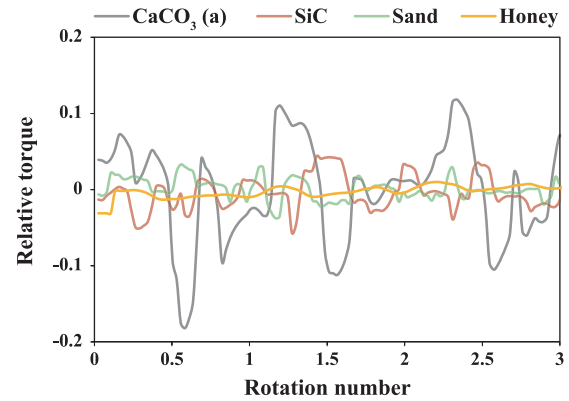


Fig. 9 Relative torque evolution $((T - \bar{T}) / \bar{T})$ at the blade tip speed = 100 mm/s.

- For all three specimens, the materials below the blade seemed to remain static.

Fig. 9 presents the relative torque measurements of the 3 powder specimens (+ honey) as a function of the number of rotations of the blade (at a blade tip speed of 100 mm/s). The relative torque is defined as:

$$\text{Relative torque} = (T - \bar{T}) / \bar{T} \quad (5)$$

where \bar{T} is the mean torque value calculated within the 50-second measurements.

The results show that the torque measurements can be correlated to the system cohesiveness:

- A cohesive granular material like CaCO_3 (a) (group C) is characterized by large and slow torque oscillations. Because of the powder cohesiveness, the powder blocks formed in front of the blade can become large before they become unstable and collapse. The “build-up/collapse” process is repeated regularly as the blade rotates in the powder bed. This leads to a periodic behavior with large events (large oscillations) and a low frequency (slow oscillations). It can be said that the “response time” is slow when the powder bed is disturbed by the blade;
- For a less cohesive powder like SiC (group A), the powder blocks become unstable and collapse earlier compared to a group C powder. This still leads to a periodic “build-up/collapse” process, but with smaller and faster events;
- For a free-flowing powder (sand, group B), the gravitational forces are preponderant compared to cohesion forces. As a consequence, no stable powder block can be formed and the “build-up/collapse” process is almost instantaneous. This leads to a continuous flow of particles around the blade. The “response time” of a free-flowing powder is fast when the powder bed is disturbed by the blade. The corresponding torque signal is quite stable and there is no periodic pattern (relatively close to the honey torque signal).

These results can be related to those obtained by

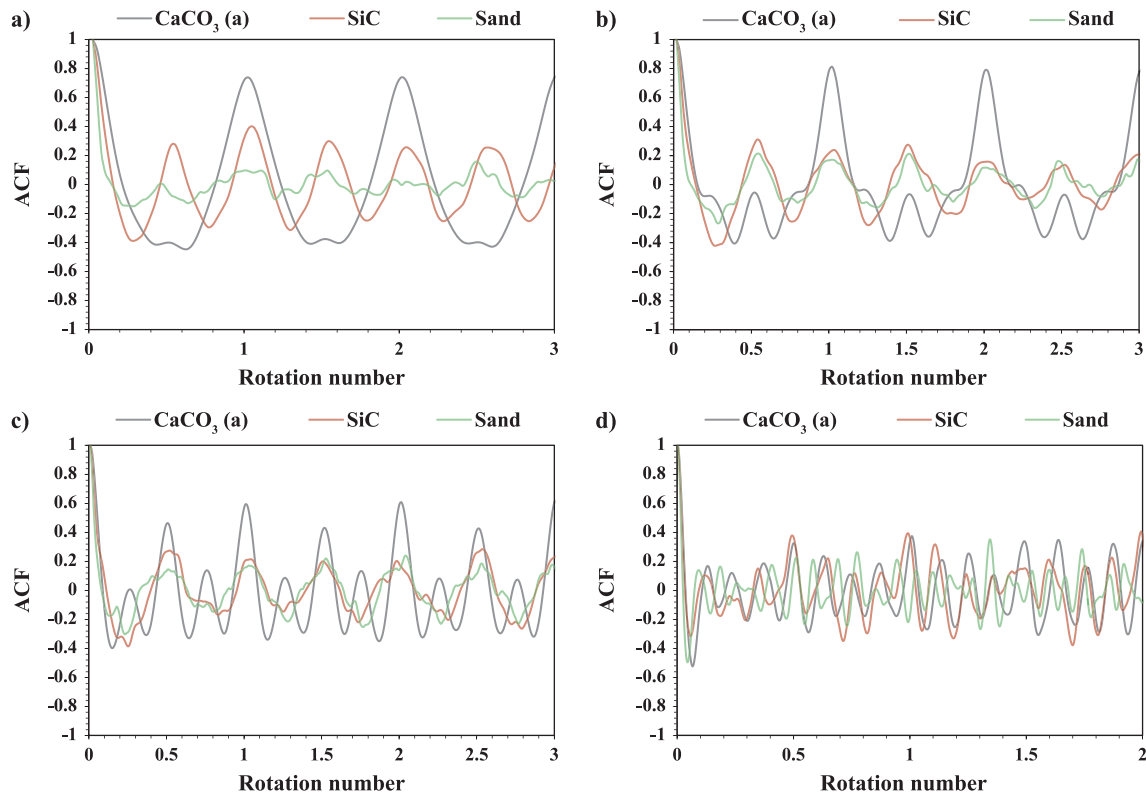


Fig. 10 Autocorrelation function at different blade tip speeds. **a)** blade tip speed = 100 mm/s; **b)** blade tip speed = 70 mm/s; **c)** blade tip speed = 40 mm/s; **d)** blade tip speed = 10 mm/s.

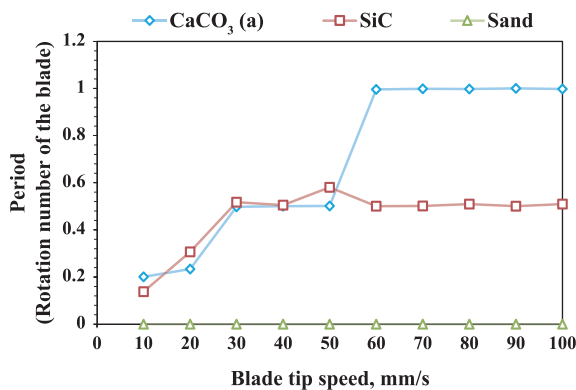


Fig. 11 Cycle time at different blade tip speeds.

Quintanilla et al. (2006), who used a half-filled and slowly rotating horizontal drum. Indeed, the collapse of the powder blocks can be assimilated to the avalanches in the rotating drum. In both cases, a periodic behavior is observed and the periodicity of the signals can be correlated to the system cohesiveness. Also, in both studies, it is shown that a higher cohesion leads to larger events (collapses or avalanches).

4.2 Periodic motion analysis and powder flowability classification

During the tests, the torque was recorded every 0.04

seconds and the 50-second measurements could then be treated as a discrete process with 1250 observations for the autocorrelation analysis. The autocorrelation functions of the three powders are presented in **Fig. 10**. These graphs indicate that the flow within the powder bed exhibits the same cyclic pattern as the flow on the surface (**Fig. 5**). However, the periodic motion on the surface shows a lower correlation level, which might be caused by the surface effects.

These ACF curves show that:

- At high flow speeds, the CaCO₃ (a) powder (group C) has a cycle time corresponding to one blade rotation and it is close to an “ideal” correlation (the maximum correlation level is close to 1). When the flow speed decreases, the cycle time of the CaCO₃ specimen is halved and the correlation level of the cyclic pattern decreases;
- The SiC powder (group A) always has a cycle time of 1/2 a rotation of the blade and a relatively lower correlation level.
- The sand powder (group B) has a low correlation level (ACF < 0.2), which means that no significant periodic motion can be detected in this case.

The cycle times (expressed in the number of rotations of the blade) of the three samples at different flow speeds are summarized in **Fig. 11**. Based on previous results, a threshold value of 0.2 was used to differentiate periodic

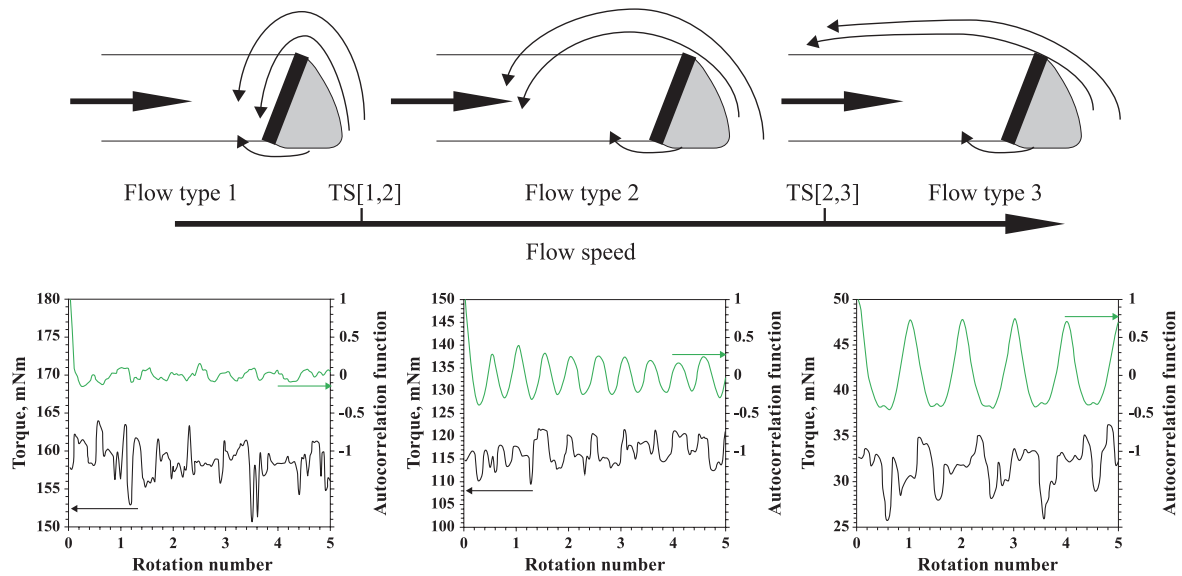


Fig. 12 Transition speeds and powder flow behaviors in the FT4 Powder Rheometer.

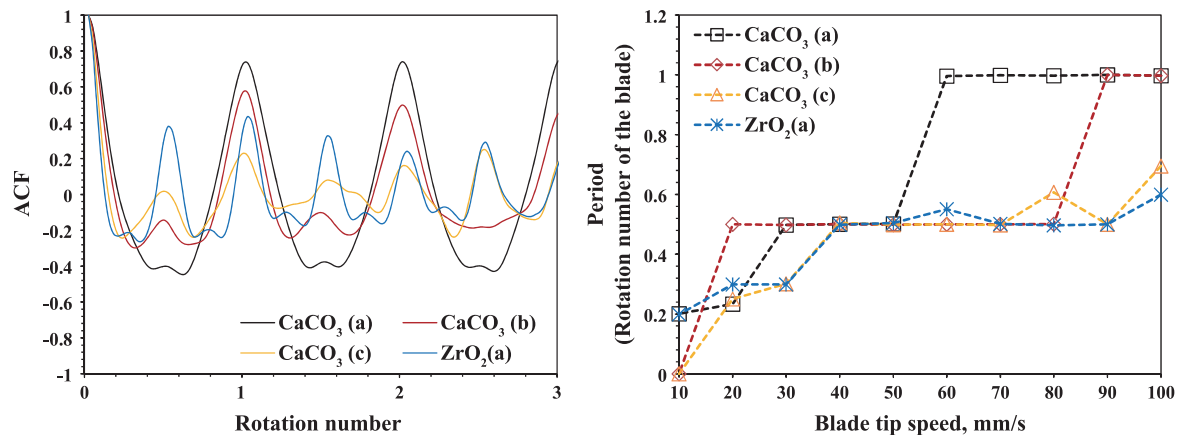


Fig. 13 Test results of the new testing method: autocorrelation function at 100 mm/s (**left**); cycle time at different blade tip speeds (**right**).

from non-periodic flow patterns. All the processes with a correlation level lower than 0.2 were ignored and considered as non-periodic flow patterns. For convenience, the cycle time of such processes was then considered as 0. The results of the autocorrelation analysis indicate that:

- The cycle time of a powder material increases as its cohesiveness increases;
- The cycle time doubles at a certain blade tip speed, which can be regarded as a “transition speed”;
- Free-flowing powders have a very low correlation level ($ACF < 0.2$) and present almost no periodic flow pattern. These powders behave almost like the liquid honey (“ideal” flow)

These results suggest that for any powder material, there are three flow behaviors:

- Flow type 1 (**Fig. 12-left**): It is observed when the response time of the powder is significantly smaller than the time required for a 1/2 rotation of the blade. As a

consequence, the particles flow freely and smoothly around the blade. The corresponding torque signal is quite stable and there are no periodic patterns. For this type of flow, the correlation level is less than 0.2 (the cycle time is set to 0);

- Flow type 2 (**Fig. 12-middle**): The response time of the powder is smaller but close to the time corresponding to a 1/2 rotation of the blade. In this case, the blade leaves a small unstable empty space behind it when passing through the powder bed. This empty space expands to a maximum volume and collapses before being reached by the other side of the blade. This type of flow corresponds to a cycle time of a 1/2 rotation of the blade and a moderate correlation level in the autocorrelation analysis ($0.2 < ACF < 0.8$);
- Flow type 3 (**Fig. 12-right**): The response time of the powder is larger than the time required for a 1/2 rotation of the blade. Thus the blade leaves a large empty

space behind it when passing through the powder bed. The other side of the blade reaches the empty space before it collapses. This type of powder flow corresponds to a cycle time of one complete rotation of the blade and a high correlation level ($ACF > 0.8$).

These results suggest the existence of two flow transition speeds (**Fig. 12**): $TS[1, 2]$ is the transition speed from flow type 1 to flow type 2 and $TS[2, 3]$ is the transition speed from flow type 2 to flow type 3. For a given powder, the values of $TS[1, 2]$ and $TS[2, 3]$ depend on its response time to plastic flow deformation. Therefore, the values of the two transition speeds can be used to identify the powder flow properties:

- Free-flowing powders such as the sand powder flow almost like a liquid and have a very short response time. The corresponding $TS[1, 2]$ and $TS[2, 3]$ values are very high. As a result, these powders exhibit the flow type 1 at any accessible flow speed with the FT4 Powder Rheometer;
- Cohesive powders such as the $CaCO_3$ powder on the contrary have a very long response time, which results in low $TS[1, 2]$ and $TS[2, 3]$ values. These powders then present all three flow types within the flow speeds accessible with the FT4 Powder Rheometer;
- The powders with moderate flowability such as the SiC powder have an intermediate response time, which means that they have intermediate $TS[1, 2]$ values (accessible with the FT4 Powder Rheometer) and high $TS[2, 3]$ values. As a consequence, these powders can exhibit flow type 1 or 2 depending on the flow speed.

Other tests were also performed to verify these conclusions. As explained in section 3.1, the standard dynamic test cannot discriminate the flowabilities between $CaCO_3$ (a), $CaCO_3$ (b), $CaCO_3$ (c) and ZrO_2 . The results of the new testing method clearly show these differences (**Fig. 13**):

- $CaCO_3$ (a) has a poorer flowability than $CaCO_3$ (b) since it has a lower $TS[2,3]$ and a higher correlation level;
- $CaCO_3$ (c) and ZrO_2 present similar flowabilities as they have similar cycle time variations. Still, $CaCO_3$ (c) flows a little more easily than ZrO_2 , as it has a higher $TS[1,2]$ and a lower correlation level.

5. Conclusion

In this study, a new flowability testing method is presented. This method is based on the FT4 standard dynamic test and quantifies the powder periodic motion under forced flow conditions. The test results prove that the dependence of the torque cycle time on the flow rate can be linked to the powder flow properties: when the

powder cohesiveness increases, the autocorrelation level and cycle time increase; conversely, a free-flowing powder tends to act like a liquid and shows no periodic motion.

This work shows that this new method has a good capability to identify and classify powder flow properties. The flowabilities of different powders which cannot be discriminated with the standard dynamic method of the FT4 Powder Rheometer are easily identified with this new method

Further studies still need to be done to optimize this method such as the influence of the blade height (30 mm in this study), the variation of the periodic motion in a wider flow speed range, as well as the influence of the blade shape.

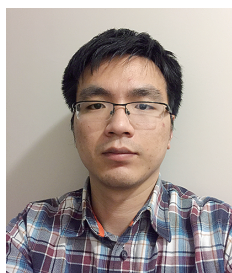
Nomenclature

BFE	Basic Flow Energy, [mJ]
$d[10]$	10 % of the population lies below the $d[10]$, [m]
$d[3, 2]$	Mean surface-volume diameter (Sauter mean diameter), [m]
$d[4, 3]$	Volume or mass moment mean (De Brouckere mean diameter), [m]
$d[50]$	Mass median diameter, [m]
$d[90]$	90 % of the population lies below the $d[90]$, [m]
E	Expected value operator
FRI	Flow Rate Index, [–]
k	Observation number in the discrete time process, [–]
n	Total observations of the discrete process
ACF	Autocorrelation function, [–]
\widehat{ACF}	Autocorrelation function of a discrete process, [–]
span	$(d[90]-d[10])/d[50]$, [–]
t	Time, [s]
T	Torque, [mN m]
\bar{T}	Average torque, [mN m]
$TS[1, 2]$	Transition speed between flow type 1 and flow type 2, [mm/s]
$TS[2, 3]$	Transition speed between flow type 2 and flow type 3, [mm/s]
X	Signal of a continuous-time process
μ	Mean
ρ	Particle density, [kg/m ³]
σ	Standard deviation
τ	Time lag, [s]

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