

## CHARACTERISATION OF COHESIVE POWDERS FOR BULK HANDLING AND DEM MODELLING

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**Key words:** Granular material, flowability, DEM, Uniaxial test, Shear test, PARDEM.

**Abstract.** The flow behaviour of granular materials is relevant for many industrial applications including the pharmaceutical, chemical, consumer goods and food industries. A key issue is the accurate characterisation of these powders under different loading conditions and flow regimes, for example in mixers, pneumatic conveyors and silo filling and discharge.

This paper explores the experimental aspects of cohesive powder handling at different compaction levels and flow regimes, namely inertial and quasi-static regimes. So far, laboratory element test set-ups capable of defining the full stress states at very low compaction levels have not been fully explored in literature. In contrast the mechanical behaviour of cohesive powders under relatively high consolidation stress (several kPa upward) can be carefully measured using element tests such as biaxial test, true triaxial and hollow cylinder tests. However in practice these tests are expensive and slow to conduct and are almost never performed for many industrial applications requiring material characterisation. Here we investigate simpler techniques that could be used for filling this important gap with the focus of providing test data for model calibration and simulation validation in line with the spirit of the European Commission funded PARDEM Marie Curie ITN Project.

We perform particle and bulk characterisation on limestone powder with 4.7 $\mu$ m and 31.3  $\mu$ m mean particle size, detergent powder with differences in formulation, cocoa powder with low and high fat content - relevant for different industrial applications. Of particular significance is the 4.7 $\mu$ m limestone powder which is the PARDEM reference powder that have been created and extensively used in a collaborative European PARDEM Project ([www.pardem.eu](http://www.pardem.eu)).

In the inertial, low consolidation stress regimes - more relevant for powder transport and conveying applications - we present experimental findings on the flowability and avalanching behaviour of the reference material in a rotating drum. On the other hand, in the quasi-static, higher consolidation regime, we perform shear tests with the Edinburgh Powder Tester (EPT), an extended uniaxial tester and the commercially available Freeman FT4 Powder Rheometer. For macroscopic quantities, we report the unconfined yield strength as a function of applied stress. These material characteristics provide important scientific insights for developing innovative solutions for cohesive powder handling problems. From these experiments and for best practice guideline, we highlight subtle issues associated with the experimental setup and measurements. The experiments lead to a rich quantitative description of the flow behaviour and failure properties of the materials which provide the material data for DEM model calibration and validation.

## 1 INTRODUCTION

Granular materials and powders constitute 75% of the material in the feedstock industries [1]. Within these industries powders are used in a broad range of processes varying from quasi-static silo filling and discharge to dynamic mixing, pneumatic conveying etc. Many storing, handling, and processing problems have been reported especially for cohesive solids. For many bulk handling applications measurement of bulk “Cohesive” characteristics provides effective solutions. An important challenge is the accurate characterisation of the “Cohesive” behaviour of cohesive powders under quasi-static and dynamic conditions.

The mechanical behaviour of cohesive powders in quasi-static regime can be carefully measured using element tests including biaxial, hollow cylinders, and true triaxial tests, however, these tests are expensive and slow to conduct and are seldom used for powder characterisation in industries. In this study we investigate simpler techniques that could be used for filling the important gap with the focus of providing test data for model calibration and simulation validation. The flow properties including unconfined strength as a function of consolidation stress was measured using Edinburgh Powder Tester, a uniaxial tester [2] and the commercially available FT4 powder rheometer [3].

For low stresses close to the surface of the bulk material, laboratory tests in rotating drum have been used for decades to understand the dynamic and shear behaviour of granular materials [4–7]. For cohesionless materials during rotation, the most evident observable property is the angle of the surface. This continuous “angle” in a rotating drum can depend strongly on the side wall which makes more it complicated to characterise such angles [8]. Other avalanching or flowability test can provide more quantities that can be used to dynamically describe and classify the behaviour of these materials. While experimental research to understand the dynamic behaviour of various non-cohesive samples has been successful, many challenges still remain for characterisation of cohesive powders [9]. Here, we perform image analysis on the experimental results obtained for cohesive samples subjected to events in a rotating drum. One goal is to find appropriate characterisation parameters for cohesive powders.

The major objectives of this paper are to present physical and flow properties of 6 different industrial powders and to classify their flow properties in quasi-static and dynamic stress regime. The repeatability of measurements from FT4, EPT, and rotary drum is also addressed for 4.7 $\mu$ m limestone powder which is the PARDEM reference powder. The relative flowability of powders in FT4 and EPT are compared. The relationships between flow properties of powders in quasi-static and dynamic states are analysed and compared.

## 2 MATERIALS AND METHODS

### 2.1 Characterization of test solids

Three pairs of samples were chosen. Each pair of samples had different levels of cohesion. The powders tested were: 4.7 $\mu\text{m}$  mean size Limestone A (commercial name ESKAL 500) and 31.3 $\mu\text{m}$  mean size Limestone B (commercial name ESKAL 30) supplied by KSL Staubtechnik, Germany; two spray dried detergent powders (Detergent A and Detergent B) with different formulations supplied by Newcastle Innovation Centre, Procter and Gamble, Newcastle, UK; and two cocoa powders with 10-12% fat (Cocoa-A) and 20-22% fat content (Cocoa-B) supplied by Nestle Product Technology Centre, Orbe, Switzerland. Please note that Limestone A is the PARDEM reference solid that have been created and extensively used in a collaborative European PARDEM project ([www.pardem.eu](http://www.pardem.eu)).

Hitachi TM 1000 Scanning Electron Microscopy (SEM) was used in this study to acquire images for visual inspection regarding shape and surface roughness of particles. The SEM images of 6 industrial solids are shown in Figure 1. Larger size Limestone B seems rougher but rounder compared to Limestone A and similar trend can be found for Cocoa B and Cocoa A. No significant difference between shape and texture of two detergent powders can be seen from the SEM images.

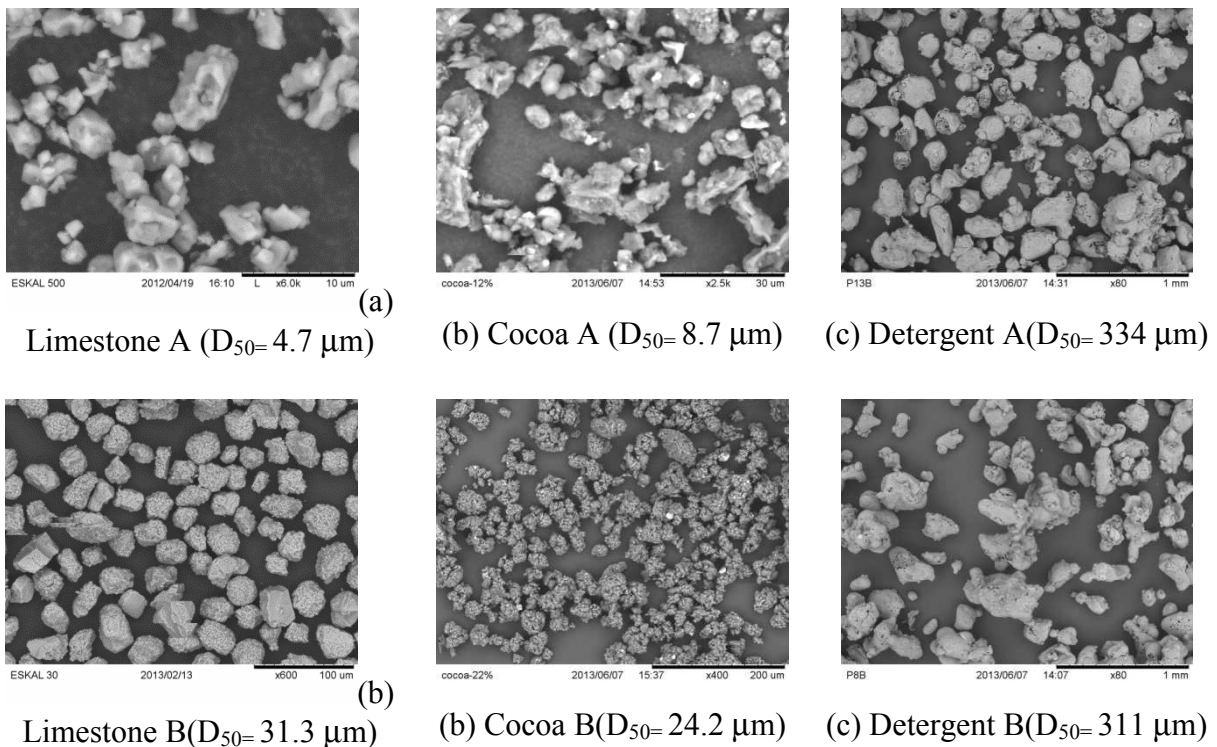


Figure 1. SEM images of industrial solids

Particle size distribution (PSD) of limestone powder was supplied by manufacturer. PSD of spray dried detergent powder was measured using mechanical sieve. The PSD of cocoa powders was measured by dry dispersion method using Malvern Mastersizer. Moisture content was measured by weighing 5 gms of a sample before and after drying in an oven at 100°C for 24 hrs. Each test was carried in duplicates. Bulk density of powder was determined by measuring the mass of the sample poured into known volume of EPT mould. The physical properties of the powders are summarised in Table 1. These powders are different in physical properties. The limestone powders are insensitive to humidity. However, detergent powders and cocoa powders are relatively hygroscopic. These samples were sealed in air tight bags and experiments were conducted in a laboratory humidity (28-45%) and temperature (18-22°C) condition.

**Table 1 Physical properties of powders**

| Material description | Fill bulk density (kg/m <sup>3</sup> ) | Mean particle size (µm) |       |       | Span=<br>$[\frac{D_{90} - D_{10}}{D_{50}}]$ | Moisture content (%) |
|----------------------|----------------------------------------|-------------------------|-------|-------|---------------------------------------------|----------------------|
|                      |                                        | D10                     | D50   | D90   |                                             |                      |
| Limestone A          | 1320                                   | 21.1                    | 31.3  | 45.83 | 0.79                                        | 0.25                 |
| Limestone B          | 754                                    | 1.42                    | 4.7   | 7.39  | 1.27                                        | 0.25                 |
| Detergent: A         | 401                                    | 167                     | 334   | 1009  | 2.52                                        | 3.38                 |
| Detergent: B         | 472                                    | 158                     | 311   | 798   | 2.06                                        | 3.06                 |
| Cocoa A              | 373                                    | 3.12                    | 8.68  | 22.5  | 2.23                                        | 5.68                 |
| Cocoa B              | 516                                    | 12.78                   | 24.23 | 47.57 | 1.44                                        | 6.14                 |

## 2.2 Flow properties measurement

The Edinburgh Powder Tester (EPT), and FT4 rheometer were used to measure flow properties in a relatively high stress (3kPa - 97 kPa) and quasi-static regime, however, rotating drum was used to measure flow properties in relatively low stress (<1kPa) and dynamic regime. The EPT (Figure 2) compression assembly consists of a cylindrical pedestal, perspex sleeve of 40 mm internal diameter, locking pin, and loading piston. The locking pin is placed in the hole in the pedestal and the sleeve rests on the pin. The Perspex is filled with powder by spoon and sample is weighed. A filter paper is placed on the top of the sample to allow for air to escape during compression. The sample is then loaded to initial stress of 7 kPa and the pin is removed. The application of initial stress generates sufficient friction between particles and wall and holds the sleeve unsupported. We propose that this action allows for two way compression of the sample and reduces the variability in bulk density across the height of the sample. The sample is then loaded to desired stress for a consolidation time of 1 minute. Once the consolidation is completed, the operator then manually slides the mould down the pedestal, exposing a free standing column of consolidated powder sample. The sample is then failed by a motor driven test piston and the stress-strain response during unconfined axial loading to failure can be recorded. The loading piston travels with a speed of 0.4 mm/s.

Friction between particle and wall is known to play an important role during confined compression of powder. The volumetric compression of the powder is reduced when the

aspect ratio of the sample is increased. In this study the effect of aspect ratio (Height to diameter ratio) on compressibility of powder was investigated using EPT. When the sample fill aspect ratio was varied in a range of 1-2, it was found that the sample bulk density increases slightly with decreasing aspect ratio, especially for Limestone A and Cocoa A. For Cocoa A the bulk density decreased only by 2.36% (COV=0.75%) when the aspect ratio was decreased from 2 to 1. The effect of aspect ratio on compressibility of the powder was not so obvious for other powder samples. This could be due to slightly higher COV in measurement of density (3.5% for Detergent A). It is important to note that the two way punching effect from EPT is an improvement over one way punching in other uniaxial tester, and it seems to allow reduction in the density variation even at high aspect ratio.

The FT4 powder rheometer (Freeman Technology Ltd., Castlemorton Common, Worcestershire, UK) was used for the flow function and effective angle of internal friction measurement in a consolidated state. The FT4 apparatus (Figure 3) is described elsewhere in literature [10,11]. The procedure used to measure the flow properties is that recommended by the standard shear technique by FT4 rheometer. Briefly, in this test, the powder was first conditioned and presheared. The conditioning involves homogenisation of sample in a 50 mm diameter cylindrical vessel by rotating a blade through the powder sample in a defined motion for 1 cycle. The vessel was then split to a volume at 85mL and loaded to a specified normal stress using a vented piston. Subsequently, the vented piston was replaced with a shearing piston and the sample was presheared at a rate of 18°/minute under the same normal stress until a constant shear stress was reached. Once the sample reached the critical state characterised by constant deformation at constant volume and constant stress, the powder sample was loaded to a normal stress lower than normal stress used during preshearing and sheared at a rate of 18°/minute again. The shear stress measured in this step defines a point on the yield locus of the compressed powder. The additional points on the yield locus are obtained by preshearing the sample again and shearing at progressively lower normal forces. The preshearing process was repeated for 4 normal stresses (3kPa, 6kPa, 9kPa, and 15 kPa) and the samples were sheared at lower normal stresses. This defined the yield locus at different preshear stresses. Further data analysis was required to derive the flow function and effective angle of internal friction. The data analysis in FT4 is automated; it applies linear fit to the points on the yield loci. The unconfined strength and major principle stresses are then obtained by drawing Mohr circles. The effective angle of internal friction is the slope of the line passing through the origin of normal and shear stress plot and tangent to the Mohr circle passing through the preshearing point. The flow function is a measure of stress needed to make an arch collapse and make the material flow and effective angle of internal friction is a measure of the friction between particles.



Figure 2 Edinburgh Powder Tester



Figure 3 FT4 Rheometer

The AeroFlow tester (TSI Incorporated, USA) was used to investigate flowability of powder in dynamic regime. The tester rotates a shallow cylindrical glass drum (125 mm diameter, 25 mm depth), containing the sample, around its horizontal axis, at a constant rate (angular velocity  $\omega = 0.3$  rpm in this study) as shown in Figure 4. When the inclination angle of the material (e.g. powder) surface becomes too great for its granular structure to support it, the powder collapses, which is referred to as an “event”. Due to the cohesive nature of the sample, an etched metal collar insert was placed around the drum’s circumferential inner wall to increase the roughness and to obtain more regular, periodic events.

The time interval between events and their (relative) amplitudes are detected and recorded by a light fixture and photo-voltaic cells assembly positioned vertically in front and behind the drum, respectively. While the original, commercial set-up with a light sensor is capable of detecting big changes, it is impossible to distinguish events. Therefore, to obtain the profiles of the powder surface, an external camera (Logitech HD Pro, Logitech Intl SA) was mounted in front of the rotating drum and images were taken in regular intervals of  $t_i = 0.25$  seconds. Measurement of the time between the events is analysed using two methods. In method I, to calculate the time between events, one needs to know when an event is deemed to have taken place. An event is said to have taken place when two criteria are fulfilled. The first criterion is difference between the angles of surface recorded for successive time-steps should be greater than 5 degrees. However due to noise and too small events we introduce an additional criteria namely that the angle of surface recorded the next 5 time-steps must be lower than the angle of surface for immediate past the event recorded. In the method II, time between consecutive events is measured by applying Fourier transformation to the raw data. The method I measures the time between consecutive events independently of the size of avalanche, while the second method measure it only for major events.

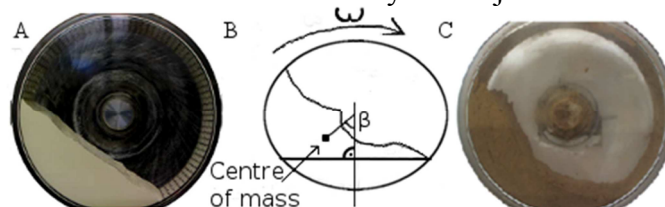


Figure 4 Angle of surface and angle of stability.

For most cohesionless samples the angle of surface is well defined (see Figure 4A). However, due to the irregular surface profile of cohesive samples (see Figure 4C), a global quantity that captures the position of the bulk sample relative to a fixed reference frame is desirable. First, to obtain the (global) surface angle, the centre of mass is needed. Every pixel in the snapshots of the drum (pixel size 6.25cm/360) is analysed along both vertical and horizontal directions. Using the pixels enables us to calculate the horizontal and vertical positions – x and y, respectively – of the centre of mass. Note that for this analysis, the powder layer sticking on the cylinder wall away from the bulk is not taken into account. From this (at least for low filling height), the angle of surface is defined as  $\beta = \text{atan}(x_c/y_c)$ , where  $x_c$  and  $y_c$  are the average values of pixels on which a powder was detected, (see Figure. 4B). The (surface) angle for a powder in a rotating drum is thus defined as the angle between vertical and the line going through the centre of material mass and the centre point of the drum. From this, the average angle (of the surface profile) can then be computed as function of time, while the maximum angle, typically measured before the events, is referred to as “angle of stability”.

### 3 RESULTS AND DISCUSSIONS

#### 3.1 Repeatability

In order to understand the uncertainties associated with the different measurement techniques repeat tests on PARDEM reference solid (Limestone A) were conducted using EPT, FT4, and rotary drum. Table 2 shows flow properties measured by FT4 and Table 3 shows the unconfined yield strength measurement from EPT. The maximum COV in unconfined yield strength and effective angle of internal friction ( $\phi_e$ ) measurement by FT4 was reported as 8.4% and 1.9% respectively. A COV of 7.4% was reported in unconfined yield strength measurement by EPT. With respect to time needed to run a flow function test with 4 preconsolidation stresses; it takes approximately 20 minutes in EPT, and 60-80 minutes in FT4 when standard testing procedure is followed.

**Table 2 Powder flow properties produced by FT4 for Limestone A ( $\mu \pm S_n$ )**

| $\sigma_p$ , kPa | UYS, kPa           | $\phi_e$ , °        |
|------------------|--------------------|---------------------|
| 3                | 1.92( $\pm 0.03$ ) | 41.85( $\pm 0.25$ ) |
| 6                | 2.52( $\pm 0.21$ ) | 39.16( $\pm 0.73$ ) |
| 9                | 3.75( $\pm 0.23$ ) | 38.02( $\pm 0.24$ ) |
| 15               | 3.97( $\pm 0.20$ ) | 37.46( $\pm 0.25$ ) |
| Maximum COV (%)  | 8.4                | 1.9                 |

**Table 3 Powder flow properties produced by EPT for Limestone A ( $\mu \pm S_n$ )**

| $\sigma_p$ , kPa | UYS, kPa           |
|------------------|--------------------|
| 17.3             | 2.50( $\pm 0.09$ ) |
| 37.1             | 3.27( $\pm 0.22$ ) |
| 56.9             | 4.70( $\pm 0.27$ ) |
| 77               | 4.97( $\pm 0.37$ ) |
| 96.4             | 5.03( $\pm 0.05$ ) |
| Maximum COV (%)  | 7.4                |

Where;  $\sigma_p$  = preconsolidation stress, UYS=unconfined yield strength,  $\mu$ =sample mean,  $S_n$ =standard deviation of sample, Co-efficient of variation (COV)= $S_n/\mu$

For rotary drum measurements, the average time between events and angle of stability for the PARDEM reference solid was found to be 8.3 sec and 53° with COV of 50.2 and 9.6%,

respectively.

### 3.2 Flow function from FT4 and EPT

The comparison of flow function from uniaxial test and shear cell test requires careful interpretation. The unconfined strength of a sample depends on the applied mean stress. Assuming axisymmetry and ignoring boundary friction, the three dimensional mean stress ( $\sigma_m$ ) in the EPT uniaxial test can be evaluated as:

$$\sigma_m = \frac{\sigma_v + 2\sigma_r}{3} \quad (1)$$

where  $\sigma_v$  is the vertical stress, and  $\sigma_r$  is the radial stress. Within the bulk solid the radial stress ( $\sigma_r$ ) is a result of applied vertical stress, which may be written as:

$$\sigma_r = k.\sigma_v \quad (2)$$

where  $k$  is lateral earth pressure ratio. Whilst the mean stress can be estimated in the EPT uniaxial test, the state of the three dimensional mean stress is not easy to evaluate in a direct shear test such as the FT4. The stress paths to failure of the samples are also different in the uniaxial and the direct shear tests. Additionally, in a direct shear test the powder is forced to fail along a predefined plane/zone whilst in a uniaxial test the sample fails along the weakest plane. Furthermore in a direct shear test, the sample supposedly reaches steady state before failure, however in EPT the sample does not reach steady state due to no pre-shearing action. Because of the aforementioned reasons it is impossible to make one to one comparison between the EPT results and the FT4 results. The measured flow function of the six industrial solids employing FT4 and EPT are shown in Figure 5 and 6 respectively.

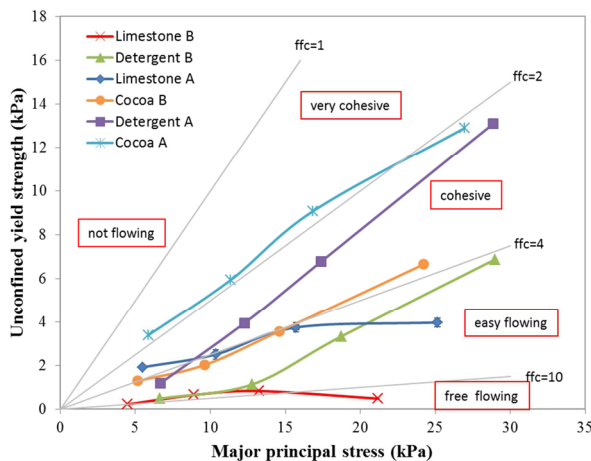


Figure 5 Flow function obtained by FT4

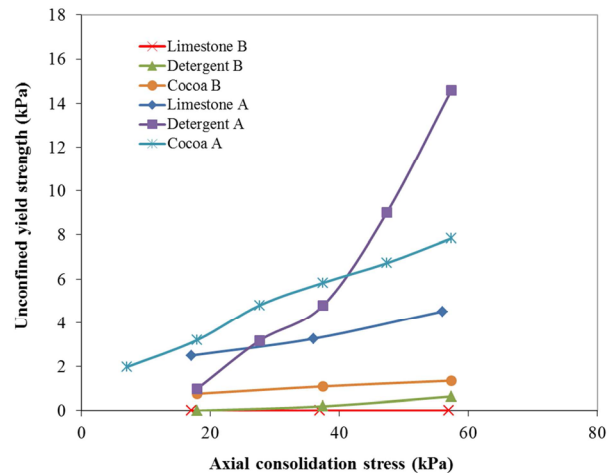


Figure 6 Flow function obtained by EPT

Powder flowability, as characterised by Jenike flow index (ffc=major principle stress/UYS) varied from very cohesive (Cocoa A) to free flowing (Limestone B) (see Figure



5). The larger the ffc better is the powder flowability. The powder flowability was found to be stress dependent. For Cocoa A, and Limestone A powder flowability generally increased with increasing consolidation stress. Most of the bulk materials exhibit such kind of behaviour and indicates elasto-plastic nature of the powders. Conversely for detergent powders, powder flowability decreased continually with increasing consolidation stress. The decrease in powder flowability with increasing stress possibly arises from plastic deformation of the soft detergent powders. For Cocoa B powder flowability first increased with increasing consolidation stress and then decreased. No trend in powder flowability with increasing stress was found for Limestone B. This could be probably due to larger scatter in UYS measurement especially at smaller strengths. No significant relationship between physical properties of the powders presented in Table 1 and flow index was found.

Table 4 shows the ranking of the flowability of the test solids using FT4 and EPT. The samples are ranked by values of the unconfined yield strength at the given consolidation stress. The powders were ranked equal when the strength was within  $\pm$ COV (measured for Limestone A). The EPT and FT4 produced similar ranking with some discrepancies. For the FT4 measurement at the major principal stress of 20 kPa, the UYS of Limestone A ranks lower than Cocoa B. In contrast, the UYS measured by EPT for Limestone A ranks consistently higher than Cocoa B. Further investigation is required to understand the reason for this discrepancy. Additionally in EPT, UYS of Detergent A at a higher stress (67kPa) is found to be higher than Cocoa A. This could be attributed to breakage and plastic deformation of detergent particles at high stress and may not be comparable to the strength at lower stresses in the FT4.

**Table 4 Ranking of powders flowability using FT4 and EPT**

| FT4, Major Principle Stress (kPa) |             |      |             |      |             | EPT, Axial Stress (kPa) |             |      |             |      |             |
|-----------------------------------|-------------|------|-------------|------|-------------|-------------------------|-------------|------|-------------|------|-------------|
| 10                                |             | 15   |             | 20   |             | 17                      |             | 37   |             | 57   |             |
| Rank                              | Material    | Rank | Material    | Rank | Material    | Rank                    | Material    | Rank | Material    | Rank | Material    |
| =1                                | Limestone B | 1    | Limestone B | 1    | Limestone B | =1                      | Limestone B | 1    | Limestone B | 1    | Limestone B |
| =1                                | Detergent B | 2    | Detergent B | =2   | Detergent B | =1                      | Detergent B | 2    | Detergent B | 2    | Detergent B |
| =3                                | Cocoa B     | =3   | Cocoa B     | =2   | Limestone A | 3                       | Cocoa B     | 3    | Cocoa B     | 3    | Cocoa B     |
| =3                                | Detergent A | =3   | Limestone A | 4    | Cocoa B     | 4                       | Detergent A | 4    | Limestone A | 4    | Limestone A |
| =3                                | Limestone A | 5    | Detergent A | 5    | Detergent A | 5                       | Limestone A | 5    | Detergent A | 5    | Cocoa A     |
| 6                                 | Cocoa A     | 6    | Cocoa A     | 6    | Cocoa A     | 6                       | Cocoa A     | 6    | Cocoa A     | 6    | Detergent A |

### 3.3 Angle of surface and angle of stability from rotary drum test

Next, we summarise the flow measurements in dynamic stage using the rotating drum device. Figure 7 shows time between event and angle of stability measurement using method I as described in section 2.2. Each point on the graph is an average value for two repeat experiments on the same sample. It can be seen that the time between events and stability angle increases as the material cohesion indicated by UYS measurement from FT4 and EPT

test increases. Additionally, the scatter of the results increased with increasing cohesion. Short and reproducible time for less cohesive powders and long and irregular time for more cohesive powder has also been reported by Kaye et al. [4] and Thalberg et al. [12]. The time between events and stability angle could not be determined for Limestone B and Cocoa B. Unlike, the other samples; we do not observe the sharp drop in the angle of surface during the experiments with Limestone B. This is due the continuous movement of the powder sample. For Cocoa B, the material stuck to the side wall of the drum which make it impossible to perform reliable measurements.

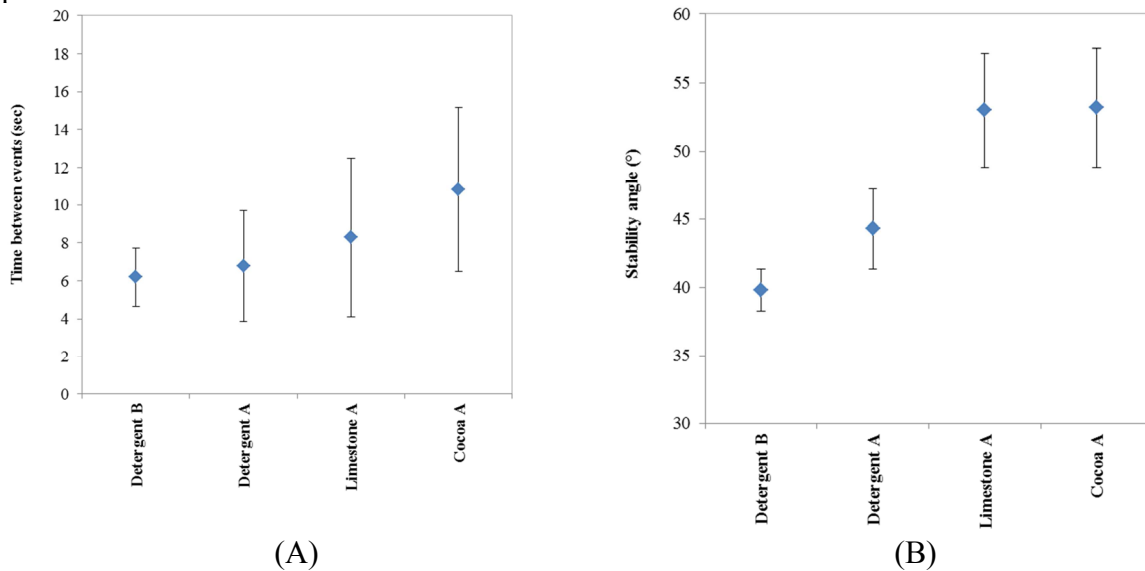


Figure 7 A) Time between event B) and angle of stability measurements from rotary drum test

The larger scatter in flow properties measurement from rotating drum makes it difficult to discriminate between flowability of different powders. The larger scatter very much reflect the characteristics of these materials at very low stress and flowing regimes where the adhesive forces lead to random formation of weak chain and agglomerate giving different structures. However, considering the mean value of measurements the powders are ranked and comparison is made with flow properties measurement from FT4 and EPT. Table 5 shows the flow properties measurement and corresponding ranking from rotary drum, FT4, and EPT.

Table 5. Measurement of flow properties using rotary drum and FT4 experiments

| Materials   | Rotating drum measurement             |                                        |                     | FT4 measurement                              |                                           | EPT measurement                             |
|-------------|---------------------------------------|----------------------------------------|---------------------|----------------------------------------------|-------------------------------------------|---------------------------------------------|
|             | Time between events (Method I)- (sec) | Time between events (Method II)- (sec) | Stability angle (°) | $\phi_c$ at 3kPa preconsolidation stress (°) | UYS at 3kPa preconsolidation stress (kPa) | UYS at 17 kPa preconsolidation stress (kPa) |
| Detergent B | 8.4 (1)                               | 6.2 (1)                                | 39.8(1)             | 42.9(2)                                      | 0.2 (1)                                   | 0 (1)                                       |
| Detergent A | 12.3 (2)                              | 6.8(2)                                 | 44.3(2)             | 44.5(3)                                      | 1.2(2)                                    | 0.98 (2)                                    |
| Limestone A | 21(3)                                 | 8.3(3)                                 | 53(3)               | 41.9(1)                                      | 1.3(3)                                    | 2.5(3)                                      |
| Cocoa A     | 25.6(4)                               | 10.8(4)                                | 53.2(4)             | 51.5(4)                                      | 3.4(4)                                    | 3.2(4)                                      |

Note: The numbers in parenthesis is the rank of powders by corresponding measured value.

The time between events estimated by both methods (method I and method II) produced the same ranking, although method II (Fourier analysis) produced larger time between events. This is obvious since method I measures the time between consecutive events independently of the size of avalanche, while method II measures this only for major events. Samples with higher cohesivity are expected to have longer time between events. The time between events ranks similar to the UYS measurement from FT4 and EPT at low preconsolidation stress. However, at higher stresses, the ranking based on UYS measurement from EPT (see Table 4) is different which reflects the stress dependency of the flowability of powders.

Samples with higher friction and cohesivity are expected to have a higher angle of stability. In this study the stability angle was found to increase with increasing cohesivity, but it do not correlate well with  $\phi_e$  alone. For example Limestone A has the lowest  $\phi_e$  and conversely the second highest angle of stability. This indicates that cohesion affects the angle of stability more than  $\phi_e$  for this specific case.

#### 4. CONCLUSIONS

This paper has summarised the flow properties for six industrial powders under different flow and stress regimes using simple characterization techniques. The physical and flow properties were measured using FT4 rheometer, Edinburgh Powder Tester (EPT), and a rotating drum device. The two way punching effect in EPT is an improvement over one way punching in other uniaxial testers; it allows reduction in the density variation across the height of the sample and increases the repeatability in unconfined yield strength measurement. Repeatability measurement on PARDEM reference solid showed that both EPT and FT4 produced repeatable measurements and can adequately discriminate between flowability of different industrial solids. The maximum coefficient of variation (COV) for unconfined yield strength measurement on EPT and FT4 was found to be 7.4% and 8.4% respectively. However, rotating drum exhibited a bigger scatter; time between events and angle of stability measurements on rotating drum had a COV of 50.2 and 9.6% respectively.

Powder flowability, as characterised by Jenike flow index, varied from very cohesive (Cocoa A) to free flowing (Limestone B). For Cocoa A, and Limestone A powder flowability generally increased with increasing consolidation stress indicating elasto-plastic nature of powders. In contrast for detergent powders, powder flowability decreased continually with increasing consolidation stress. The decrease in powder flowability with increasing stress possibly arises from plastic deformation of the soft detergent powders. For Cocoa B powder flowability first increased with increasing consolidation stress and then decreased. No trend in powder flowability with increasing stress was found for Limestone B. This could be possibly due to larger scatter in UYS measurement especially at smaller strengths. Regarding the ranking of powder by EPT and FT4, both experiments produced very similar ranking with some discrepancies when the samples were ranked by values of the unconfined yield strength at the given consolidation.

The larger scatter in flow properties measurement from rotating drum makes it difficult to discriminate between flowability of different powders. The larger scatter very much reflect

the characteristics of these materials at very low stress and flowing regimes where the adhesive forces lead to random formation of weak chain and agglomerate giving different structures. When considering the mean values, the time between events and the angle of stability from rotating drum were found to increase with increasing unconfined yield strength measurement at the low stresses. This suggests that the time between events is an indicator of cohesion; short and reproducible time indicating less cohesion and long and irregular time indicating larger cohesion.

The experimental results have provided the test data for DEM model calibration and simulation validation in line with the goals of the European Commission funded PARDEM Marie Curie ITN Project.

## **5. ACKNOWLEDGEMENTS**

The support of the European Community under the Marie Curie Initial Training Network for the PARDEM Project is gratefully acknowledged. The authors would also like to thank Prof. Stefan Luding for useful discussions.

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