Citation: Berry, R.J., Bradley, M.S.A. and McGregor, R.G. (2015) Brookfield powder flow tester - Results of round robin tests with CRM-116 limestone powder. Proceedings of the Institution of Mechanical Engineers, Part E: Journal of Process Mechanical Engineering, 229 (3). pp. 215-230. (doi:10.1177/0954408914525387)

Brookfield Powder Flow Tester - Results of Round Robin Tests with CRM -116 Limestone Powder

R.J. Berry[1], M.S.A. Bradley[1], & R.G. McGregor[2]

1 The Wolfson Centre for Bulk Solids Handling Technology
University of Greenwich
Central Avenue, Chatham Maritime, Kent, ME4 4TB
Tel: 0208 331 8646
r.j.berry@greenwich.ac.uk

2 Brookfield Engineering Laboratories, Inc.
11 Commerce Blvd., Middleboro, Massachusetts 02346 U.S.A
Tel: 508-946-6200

Abstract

A low cost powder flowability tester for industry has been developed at The Wolfson Centre in collaboration with Brookfield Engineering and four food manufacturers: Cadbury, Kerry Ingredients, GSK and United Biscuits. Anticipated uses of the tester are primarily for quality control and new product development, but it can also be used for silo design.

This paper presents the preliminary results from Round Robin trials undertaken with the PFT using the CRM -116 limestone standard test material. The mean flow properties have been compared to published data found in the literature for the other shear testers.

Keywords

Shear cell, crm 116 limestone powder, flow function, Characterizing powder flowability, reproducibility, Brookfield Powder Flow Tester, Jenike Shear Cell, Schulze Ring Shear Tester

1.0 Introduction

The Brookfield Powder Flow Tester, launched in January 2010, was developed through a Defra sponsored collaboration between The Wolfson Centre for Bulk Solids Handling Technology, viscometer manufacturer Brookfield Engineering and food manufacturers, Cadbury, GSK, Kerry Ingredients and United Biscuits. The aim of this work was to develop a powder flowability tester that gave demonstrably meaningful results, was quick and easy to use in trained but unskilled hands, presented data in a manner that was easy to interpret by non-powder specialists and was relatively economical to buy, at the time approximately £10K. The machine was developed around an automated annular shear cell, following Jenike silo design principles [1]. The key difference though was that, while the machine could be used to undertake a silo design, the main intended uses were to quantify the flow properties to assist with:

- Process and product improvement
- New product formulation
- Comparing flow of new versus old ingredients
- Quality control on incoming or outgoing batches
- Application to minerals, chemicals, food, pharmaceuticals, cosmetics, etc.

The development of the machine took approximately 5 years during which time numerous papers were published relating to the optimisation of the shear testing procedure [2&3], cell geometry [4] as well as the results of industrial trials to demonstrate the usefulness of the machine [5&6].

1.1 Objective

The objective of this paper is to demonstrate the reproducibility of measurements made by the production version of the Brookfield Powder Flow Tester (PFT) and compare the magnitude of the flow property measurements it generates to other commonly used shear testers.

To achieve this, round robin tests have been undertaken with PFT using the standard BCR limestone powder that was originally made available for Jenike shear tester operators to compare their testing technique to a standard set of test data published by Akers [7]. At present round robin tests have been undertaken with 3 laboratories', Brookfield Engineering Laboratories (US), Brookfield Viscometers Ltd (UK) and The Wolfson Centre for Bulk Solids Handling Technology (UK), with 7 different machines and 7 different operators. For each machine 7 repeat flow functions have been measured for both the standard and small volume shear cells, thus giving a total of 49 repeat tests for each cell size. Brookfield is currently in the process of expanding the testing to established customers.

In addition to the standard Jenike shear cell data, a review of the literature demonstrates that of the current commercially available shear testers, the Schulze RST [8] and Freeman FT4 Powder Rheometer with shear cell attachment [9], similar round robin tests have been undertaken for the former [10]. Limited shear tests undertaken with the BCR limestone using the Freeman FT4 shear cell have been published by Freeman [11]. Limited shear tests with industrial machines that are no longer commercially available such as the Peschl [12] and Johanson Indicizer [13] using BCR limestone have been published by Bell [14] as well as Uniaxial unconfined failure test data by Enstad [15].

2.0 Overview of The Brookfield PFT

The production Brookfield Powder Flow Tester that was used for the trials is presented in figure 1a, with the standard volume shear cell (263cc) and filling accessories in figure 1b. The Brookfield tester has been designed to minimise the operator involvement in the testing process. The only operator involvement in the testing is attaching the required lid to the compression plate, filling powder in the trough, and loading the filled cell onto the drive of the machine. The machine is computer controlled via a USB link using Powder Flow Pro software.

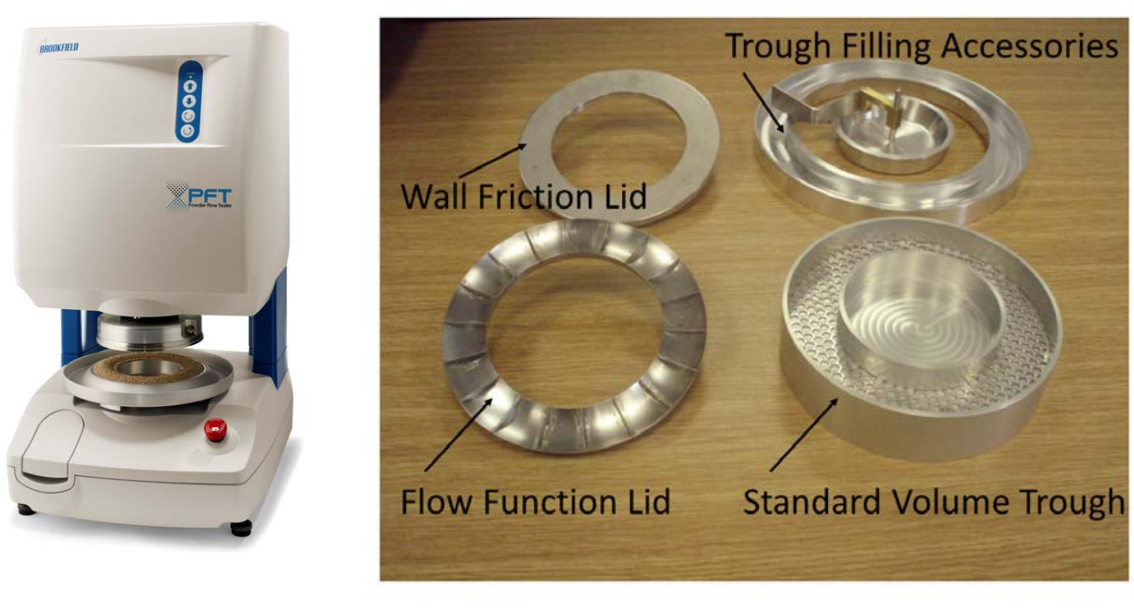
As a brief overview the machine operates four basic tests (the first of which is the focus of this work), namely:

1. **Flow Function**; this is a measurement of the internal resistance to flow of a powder, often manifested in its ability to form a blockage (usually an arch or a rat-hole) in a hopper or feeder.

2. **Time Flow Function**; this is similar to the Flow Function but characterising the ability of the powder to gain strength when left in static storage for a period of time, often leading to powder flow being hard to start up after a shut-down.

3. **Wall Friction**; the friction developed between the powder and a constraining surface, which controls the flow pattern that forms when a vessel discharges as well as the tendency for the powder to flow or hang on the surface of a chute. A long travel wall friction test allows the user to investigate the evolution of wall friction (section 3.4) with increasing shear distances over the wall.

4. **Bulk Density and compressibility**, which particularly affect the ability of a powder to pack into bottles, boxes or other packages of given size; this is also used in some quarters as an outline indicator of powder flowability. Note that bulk density measurements are all produced by the Flow Function and Wall Friction tests, however, if only bulk density information is required, the Bulk Density test is a quicker one.



a) *b)*

Fig 1, Brookfield Powder Flow Tester a) Machine and b) Shear cell troughs, lids and filling accessories

The speed of the machine is such that a full Flow Function test can be undertaken in around 35 minutes for the standard (5 consolidation level) test, although a short-cut test (2 consolidation level) for quick comparison of samples can be undertaken in as little as eleven minutes.

The economical sales price has been achieved, with the list price of the machine being around a quarter of any comparable flow property tester previously on the market.

3.0 Experimental Method and Preliminary Results

The BCR limestone standard for the Jenike shear cell [7] provides flow property measurements at consolidation normal stresses of 3, 6, 9 and 15kPa. For the round robin test undertaken in this work, it was not possible to test at the same stress because the:

- Brookfield PFT operates over a significantly lower consolidation normal stress range (0.3 to 5kPa) in the case of the standard volume cell ,
- rigid test structure, agreed upon for ease of use, limits the ability of the operator to customise the consolidation stresses, and failure stresses (at present)

Therefore the round robin tests for the Brookfield PFT were undertaken at 5 consolidation normal stresses geometrically spaced over the full range of the machine for both the standard and small volume shear cells. (Note that preliminary trials presented in section 4.0 examined both geometric and even spacing of the consolidation levels but found the former to be more satisfactory) At each consolidation level two failure points were measured. These consolidation and failure stresses are summarised in table 1 below. A more detailed explanation of the test algorithm and locus construction is presented below in section 3.3-3.4. The spacing of the multiple consolidation stresses used can either be geometric or even.

Cell size	**Standard volume**					**Small volume**				
Locus number	1	2	3	4	5	1	2	3	4	5
Consolidation Normal Stress σ_E [Pa]*	289	584	1180	2385	4819	795	1607	3246	6559	13252
Over-consolidation failure stress 2 [Pa]*	193	393	787	1590	3213	530	1071	2164	4373	8835
Over-consolidation failure stress 1 [Pa]*	96	195	393	795	1606	265	536	1.082	2186	4417

Table 1, Summary of the consolidation normal stresses and over-consolidated failure stresses used in the standard and small volume cell tests

*These are the normal stresses applied to the lid. The actual stress on failure plane is higher due to self-weight of powder in the cell lid approximately 35Pa and 19Pa in the standard and small volume cells respectively at the highest consolidation level.

Regarding the stress range of the PFT, the device was developed in conjunction with the food industry where most of the powders being handled where below 800kg/m^3 bulk density. It was developed for the purpose of quality control and formulation primarily, so the low stress flow behaviour was the key concern. Nethertheless, applying the ASTM [16] guidelines for the consolidation stress range for silo design for bulk solids below 800kg/m^3 requires a lower level of 2kPa, which is driven more by the lower limit of the Jenike tester rather than of this being the useful lower limit. The recommendation for silo design is test to an upper limit of 8 times the initial consolidation level of 16kPa. With the small cell this range can be approximately covered with an additional low stress measurement at half required lower limit with the standard flow function test (see table 1) which has the following progression of consolidation normal stresses: 0.85, 1.6, 3.2, 6.5, and 13kPa. For higher density materials 1800 to 2400kg/m^3 the standard test with the small cell will cover significantly below the recommended lower limit of 3kPa, and run to a maximum of just over 4 times this value.

3.1 Sample preparation and cell filling

All tests were carried out on fresh samples of BCR limestone that had been equilibrated to 20-25˚C, 40-60%RH for 48hours prior to testing. Since the filling of the powder into the trough is the area where the operator can have the greatest influence on the result, care was taken during sample filling. The objective of trough filling is to produce a homogenous bed of material that has been consolidated under only its self weight.

For the standard volume cell (25mm wide trough) the bulk solid was allowed to flow under gravity from a low height into the trough using a standard 5cc metal scoop. The inner & outer catch trays were used in conjunction with the rotating levelling tool (see fig 1b) to ensure a repeatable fill volume. For the tests on the 43cc small volume cell with only a 10mm wide trough it was necessary to brush the powder into the trough through a coarse 1mm sieve to break up agglomerates and minimise voidage; nevertheless, this still resulted in lower fill density for the small volume cell as shown in table 2. Preliminary tests, where the operator attempted to scoop powder into the small trough, resulted in an even lower fill density and excess compaction at the high stress end of the test.

3.2 PFT flow function test algorithm

While the majority of the commercially available shear testers [7, 8, & 9] undertake flow function measurements at a single consolidation stress level per powder sample, the standard test algorithm for the Brookfield PFT undertakes failure property measurements at five increasing consolidation stress levels over the range of the machine. Note that this approach was driven by experience of industrial clients who were either trying to characterise their material at a single consolidation level, or did not bother to measure the flow properties at all because of the excessive testing time required to use a fresh sample for each test. The multiple measurement approach does not appear to significantly reduce the indicated strength for most materials (as demonstrated later for BCR limestone) and has several benefits:

- all measurements are attained from a single cell filling, reducing sample to sample variations,
- a reduced testing time; 7 minutes to fill/empty the cell, plus 28 minutes for a 5 point flow function gives a total testing time of 35 minutes. Using separate samples for each consolidation level would require an additional 28minutes (4 x 7 minutes) spent filling and emptying the trough.
- ease of processing and interpreting data as a full flow function can be generated from a single test.

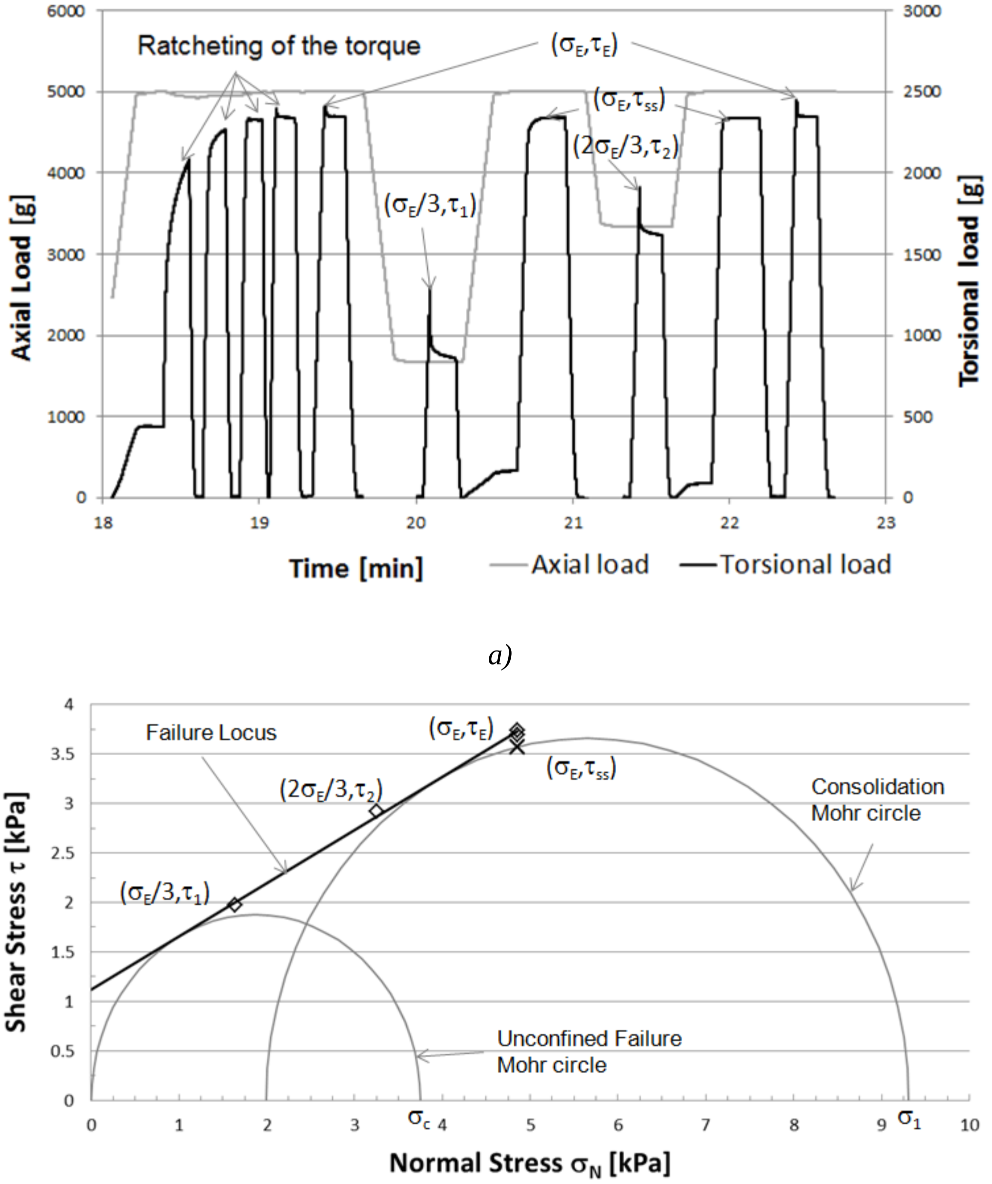
At each new consolidation stress level, the philosophy of the control algorithm is to take all failure measurements quickly within a minimum shear displacement whilst the shear stress is close to the peak (simple shear) before the powder breaks into two blocks (pure shear) and the shear stress stabilises to a lower value [2]. When the consolidation stress is increased to the next higher level, it is assumed that the doubling in normal stress applied is sufficient to negate any previous stress history. The results of tests with BCR limestone to evaluate the affect of using a single sample for all 5 stress levels versus using fresh samples at each stress level are presented in section 5.1.

3.3 Achieving repeatable consolidation

To achieve a repeatable consolidation prior to failure locus measurement, the PFT control algorithm ratchets the shear stress, by rotating forward (0.5° rotation) and backward (to reduce torque to zero) under the consolidation normal stress until the peak shear stress stabilises to within a defined limit, as shown in fig 2a below. To ensure the test completes, a maximum of 15 ratchets are run before critical consolidation is assumed and the failure loci are measured as below. During the development of the machine a number of different algorithms were trialled, including shearing for a large fixed displacement (e.g.10 and 20° rotation), or running a fixed number of re-shears (2 and 4) at the consolidation normal stress. In summary the ratcheting approach achieved repeatable consolidation in a shorter time than the continuous shearing method, but generates a shear stress peak at consolidation normal stress.

3.4 Failure locus construction

The method of locus measurement used by the Brookfield PFT shown in fig 2b differs from the ASTM D6128 standard for the Jenike shear cell [16] in one minor detail only. The point ‘e’ is not considered the end point of the locus. That is because of the inclusion of the peak shear stress τ_e at the consolidation normal stress σ_e in the failure locus. While this is somewhat unconventional, it has been used successfully at The Wolfson Centre in various annular testers [17] over many years. A review of the literature shows that other researchers Peschl [18 & 19] and Hohne (as described by Schulze, p151 [20]) have recognised and used this technique.



a)

b)

Fig 2, Example flow property measurements for BCR limestone at 4.8kPa normal stress a) Ratcheting of the shear stress to ensure critical consolidation (where axial load is in red and torque is in blue) and b.) Construction of the failure locus and determination of the principal stresses

Thus the procedure used with the PFT for the construction of the locus is as follows; A locus is constructed from peak shear stresses measured at the consolidation normal stress, then at 1/3 and 2/3 of this value, and followed by a final re-shear at the consolidation normal stress to check for consistency with the first peak. These values were tabulated in table 1. A best fit linear failure locus is then determined from the 4 data points. The unconfined failure strength σ_c is determined from a Mohr circle that touches the locus tangentially and passes through the origin. The major principal consolidation stress σ_1 is determined from a Mohr circle that touches the locus tangentially and passes through the average steady state stress point. This is determined from the two reconsolidation steps after the over-consolidated failure measurements.

The inclusion of the consolidation normal stress point in the failure locus gives a wide spread of measurements for linear extrapolation into the low stress region. In practice the peak stress at the end point and point of tangency are low relative to the best fit linear trend through the data. Although most loci have a slight curvature, the algorithm only allows for a linear interpretation, as it is assumed that a linear fit that is consistently in error, gives better reproducibility than a curve fit that picks up natural scatter in the data measurements.

4.0 Preliminary results

Preliminary trial using two machines were undertaken to compare the effect of using geometric versus even consolidation stress increases on a single powder sample. It was assumed that the increase in consolidation stress was sufficient to compact the powder, with the vanes of the lid cutting a new failure plane, and return the sample to a pure shear condition at the start of each new consolidation level. The results of this comparison are presented in fig 3, based on the mean flow functions from 7 repeat tests on two PFTs, for both the standard and small volume cell. The comparison shows that the even spacing resulted in slightly lower strengths at the higher consolidation stresses for both cell sizes but the difference was not significant. It was suspected that the proportionately smaller increases in consolidation were less effective at removing the previous stress history causing a reduction in strength. Thus the geometric range was selected for the full testing because it made use of the full stress range of the machine and gave higher failure strength values.

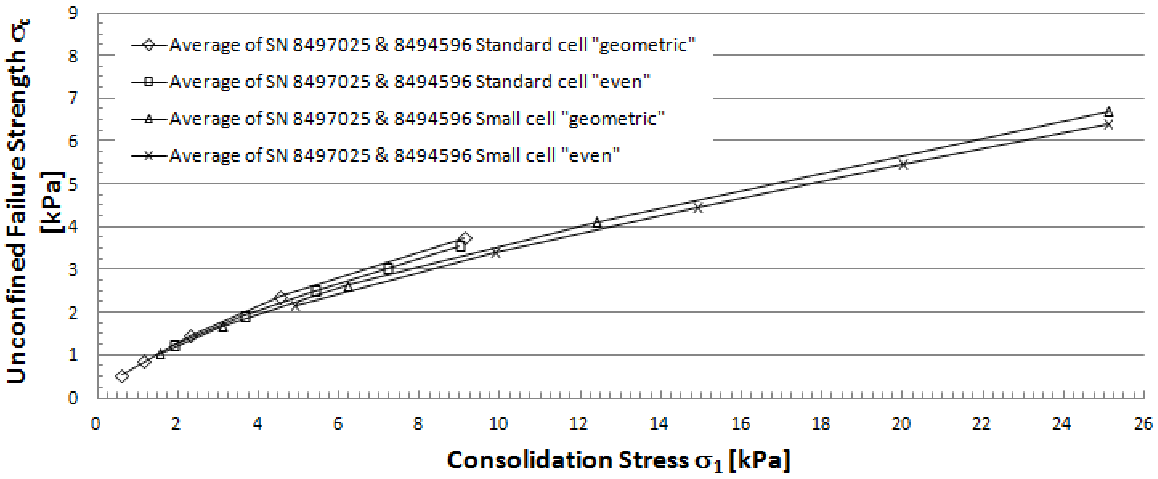


Fig 3, Flow function tests with two machines comparing geometric versus even spacing of the consolidation normal stresses

Thus for the full trials a custom flow function test was run for each cell size, operating over the full stress range of the machine using 5 consolidation stresses in a geometric progression. Three over-consolidation points were used at each consolidation level. These test stresses are summarised in Table 8.1. Note that each 5 point flow function was generated from a single shear cell filling. Therefore the total test time was approximately 24 minutes, with all 7 repeat tests easily completed within a working day.

5.0 Brookfield PFT Results

The results of the repeat flow function tests are presented by machine serial number in fig 4a&b for the standard and small cells respectively. These show good grouping of the derived points; differences are generally due to all data points drifting up or down between repeat tests, rather than repeated crossing of the flow functions.

The mean data from 7 Brookfield PFT, 7 repeats each are presented in Table 2 and figs 5a,b&c as flow functions, internal friction functions and bulk density functions respectively. The error bars represent the standard deviation at 95% confidence interval. Inspection of the flow functions shows that the standard cell measures a slightly higher strength than the small cell, but the internal frictions are comparable. Inspection of the bulk densities shows that the small cell has lower initial fill density; it is suspected that this is due to difficulties when filling the narrow width of the small cell which leads to excessive voids; over the 2 to 4kPa consolidation stress range, the bulk densities are consistent between the cells. Above 4kPa the small cell measures a higher bulk density; this is to be expected because the proportionally larger radial clearance gap allows more powder to be expressed from the cell and therefore lost to the measurement.

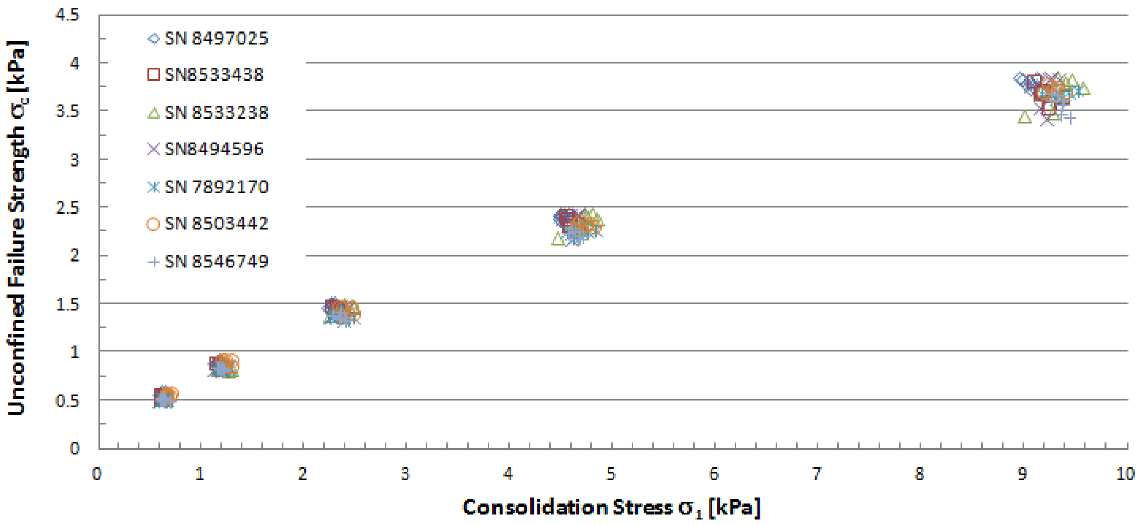
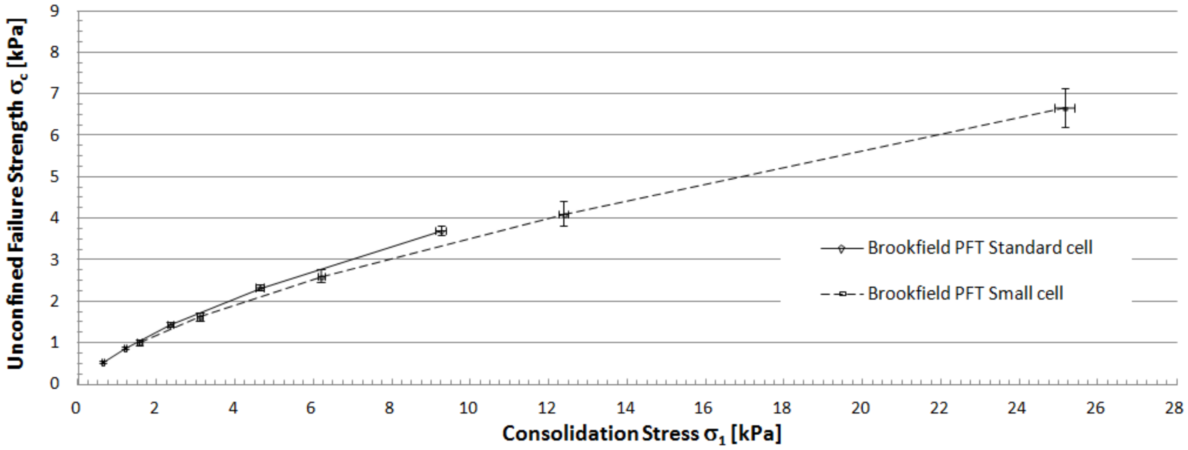


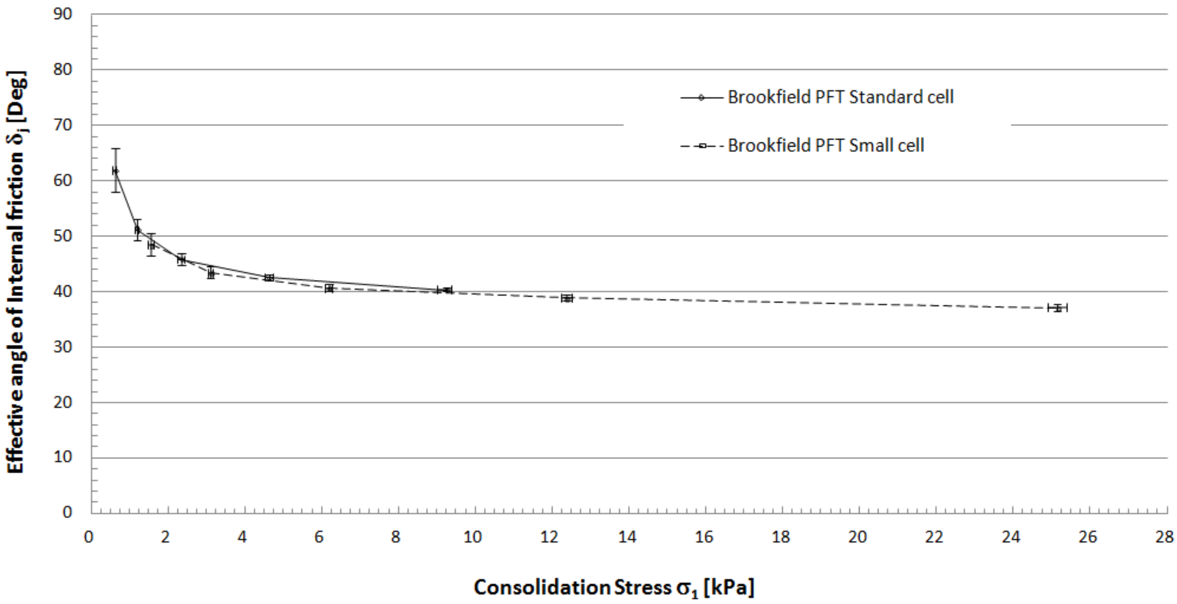
Fig 4, Comparison of the flow functions by machine serial number, a) Standard volume cell & b) Small volume cell

Standard Volume Shear Cell (263cc)					**Small Volume Shear Cell (43cc)**				
σ_E [kPa]	σ_1 [kPa]	σ_c [kPa]	ρ_B [kg/m^3]	δ_j [deg]	**σ_E [kPa]**	σ_1 [kPa]	σ_c [kPa]	ρ_B [kg/m^3]	δ_j [deg]
-	0.079	-	618.3	-	-	0.032	-	558.1	-
0.310	0.624	0.539	759.2	61.86	**0.801**	1.552	1.01	854.5	48.50
0.609	1.194	0.867	833.6	51.14	**1.617**	3.098	1.635	954.4	43.41
1.209	2.343	1.436	919.0	45.79	**3.260**	6.188	2.604	1074.5	40.60
2.417	4.630	2.329	1010.3	42.53	**6.574**	12.373	4.109	1220.6	38.86
4.854	9.245	3.702	1104.4	40.35	**13.269**	25.151	6.666	1441.0	37.14

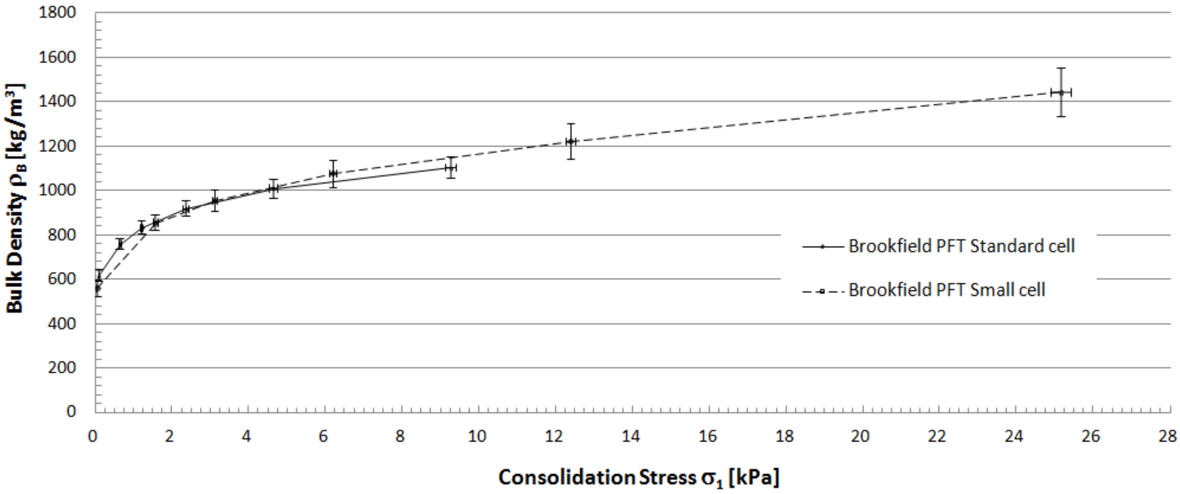
Table 2, Average flow properties for BCR limestone measured with the Brookfield PFT



a)



b)



c)

Fig 5, Comparison of the average flow properties measured for the BCR limestone in standard and small volume shear cells a) flow functions, b) effective angles of internal friction function & c) bulk density function

5.1 Effect of using single sample per consolidation level versus multiple consolidations on a single sample

A limited run of 14 tests were undertaken on three machines (in three different labs) using the standard size cell, where a fresh sample of conditioned BCR limestone was used at each new consolidation stress level and average data determined. This has been compared with mean flow function obtained from the 42 tests using the single sample per in figure 6. Inspection shows that using a fresh sample makes no significant difference for the BCR limestone with possible exception of the highest consolidation stress level where the strength increased by approximately 0.1kPa.

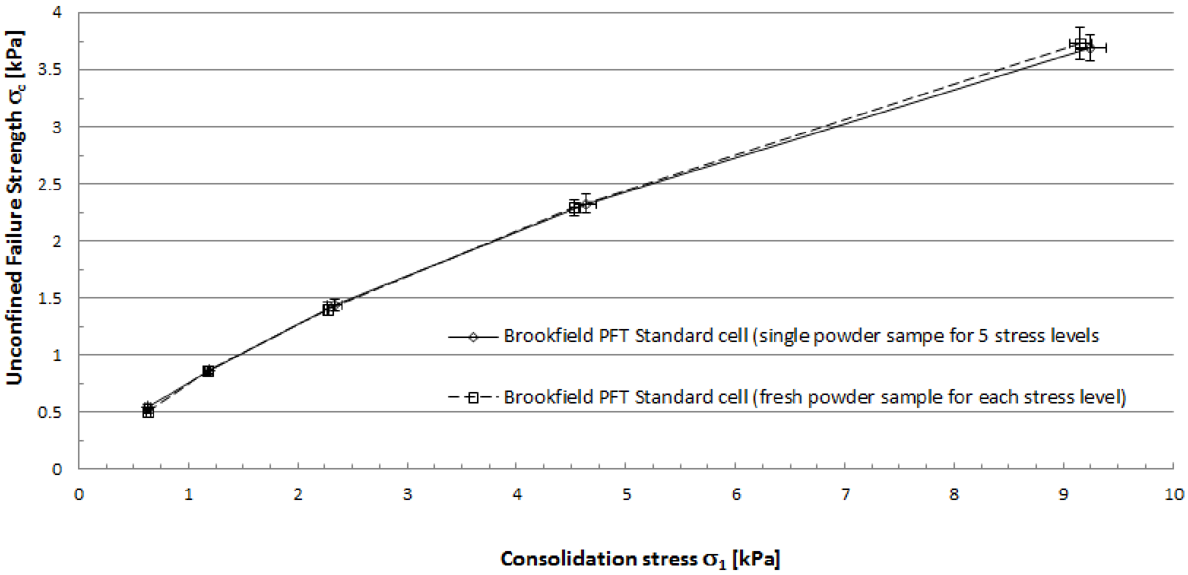


Fig 6, Comparison average flow functions measured for BCR limestone using a single sample per consolidation stress level versus a single sample for all 5 consolidation stress levels

6.0 Comparison between the failure loci of the Brookfield PFT and other testers

It was not possible to compare the Brookfield PFT failure loci, with published, data from round robin trials with the Jenike [7] and Schulze [10] testers because of differences in the operating stress ranges of the machines.

The preliminary tests undertaken with two PFT's using the standard volume cell with even spacing of the consolidation stress resulted in loci generated at 3kPa consolidation, which is the lowest stress level used in the BCR standard [7]. Thus a direct comparison was possible at this stress level for limited data as presented in fig 7, where loci represent the mean with lines at the bottom of the graph indicating the standard deviations of the respective instruments. The Brookfield PFT locus lies slightly above the mean loci of the Jenike, but below the loci from the two Schulze cells, which also have steeper gradients. Note that the Brookfield failure locus is constructed differently to the Jenike and Schulze as detailed earlier in section 3.4, due to the inclusion of both a peak and steady state stress point at the consolidation normal stress. The mean steady state value for the Brookfield is close to the pre-shear point for the Jenike.

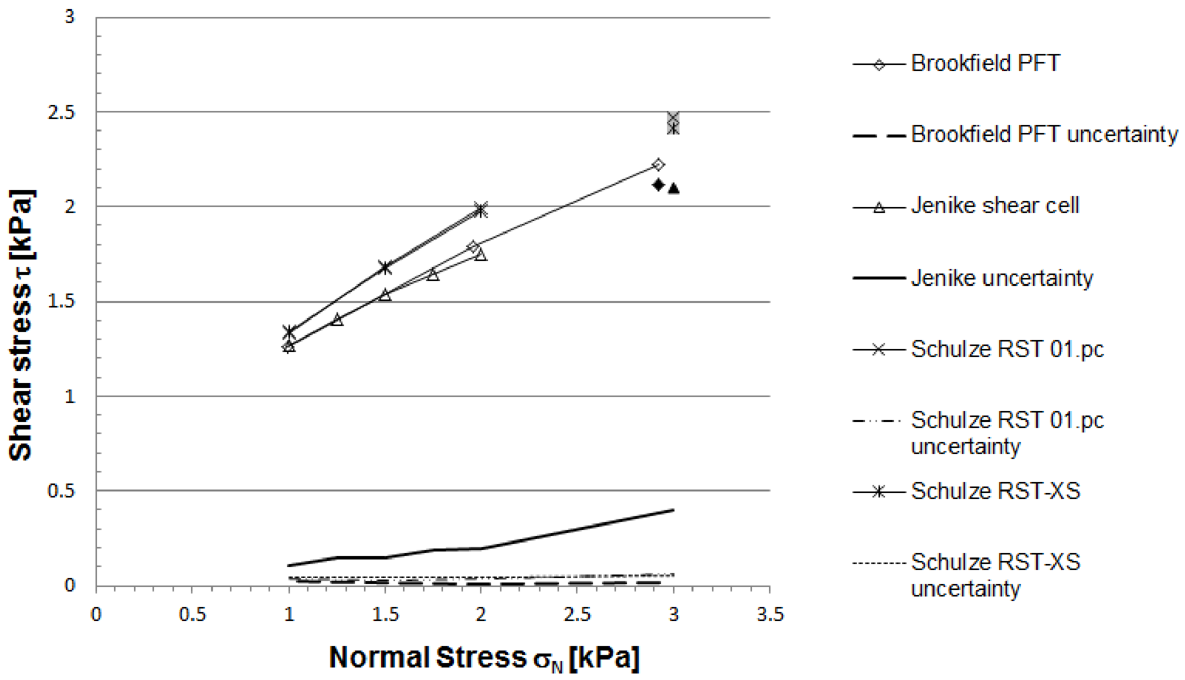


Fig 7, Comparison of the mean 3kPa failure loci measured for BCR limestone in different shear testers

For the full data determined for the Brookfield PFT none of stress levels used correlated with the BCR limestone standard as shown in fig 8. Here the 5 mean loci from the Brookfield standard volume cell are compared with the 3 & 6kPa mean loci for the Jenike and Schulze cells. The standard deviations have been omitted from the comparison for clarity but would show that scatter in the Jenike data is significantly greater than the torsional testers (as in fig 7). At the 6kPa consolidation level, deviation is 400Pa for the Jenike, 100Pa for the Schulze and 40Pa for the Brookfield (at 5kPa consolidation). To provide a comparison of the measurements, linear regression has been used to determine trend lines for the:

- loci of end points (i.e. the loci of the steady state or pre shear points),
- failure loci measured from all machines at all stress levels so the gradients and intercepts could be compared.

The mean loci of end points determined from the round robin tests on the different machines are presented in figure 9 below. The loci of end points show that the Jenike data has significant scatter and a 0.06kPa negative intercept with the shear stress axis which is clearly in error. The Schulze and Brookfield data indicate less scatter and intercepts of the order of 0.27 and 0.1kPa respectively. The gradients of the Brookfield data are comparable with the Jenike but shallower than the Schulze.

The linear gradients and intercepts of the failure loci measurements are compared in figs 10a&b respectively. Inspection of the gradients (fig 10a) shows that the Schulze loci are broadly steeper over the stress range tested, but there is a discrepancy between the large and small cell, the latter giving a shallower gradient. The Jenike data shows the lowest gradient and also shows a strong reduction in slope with reducing stress and falls significantly below others machines at 3kPa. The Brookfield PFT shows gradients between the Jenike and the Schulze in the region where data overlaps (but is closer in magnitude to the former). It also shows a strong reduction in slope as the consolidation stress tends to zero. However if the Brookfield data is interpreted in the same manner as the Jenike and Schulze, i.e.

ignoring the failure locus point at the consolidation normal stress as illustrated by "PFT method 2" in figs 10a&b, the gradient is approximately midway between the other two machines.

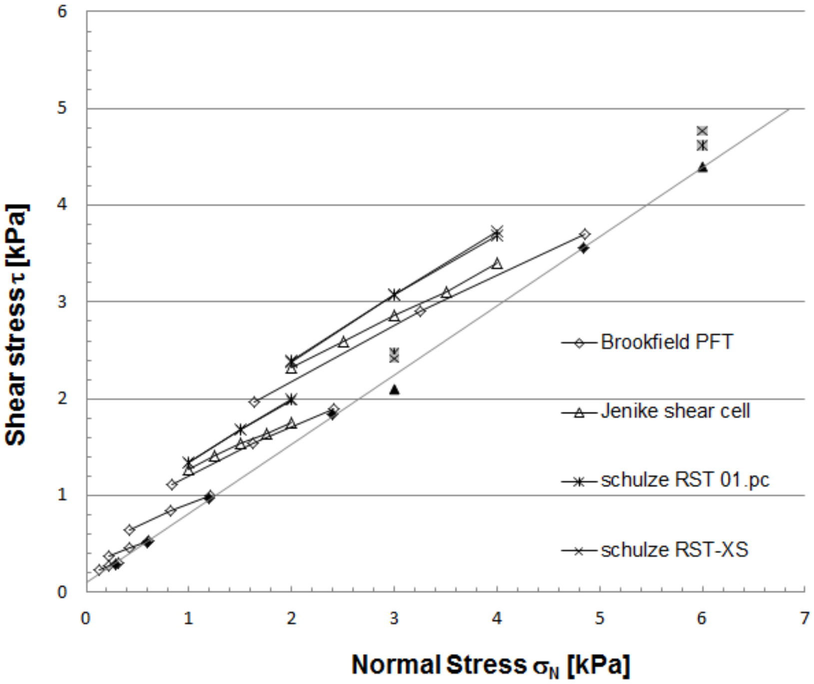


Fig 8, Comparison of the failure loci measured for the BCR limestone with the Jenike, Schulze and Brookfield shear cells from round robin tests

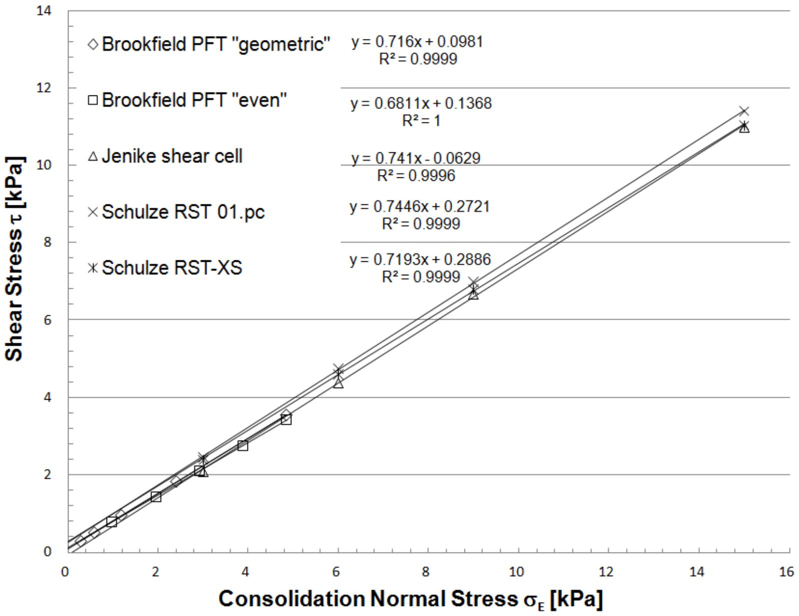
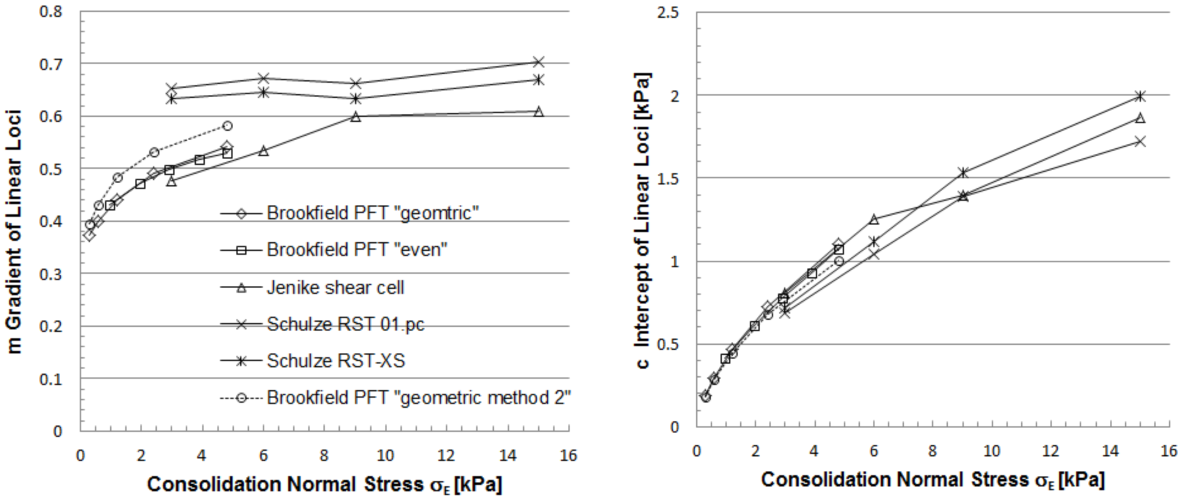
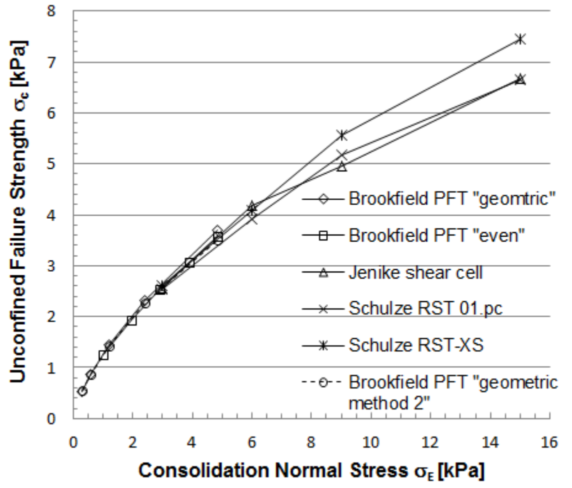


Fig 9, Comparison of the loci of end points measured for BCR limestone for the Jenike, Schulze and Brookfield shear cells from round robin tests.



a)

b)



c)

Fig 10, Comparison of mean loci data from round robin tests with BCR limestone, presented as a function of consolidation normal stress, a) gradient of best fit locus (through all data points), b) intercept of linear locus with shear stress axis & c) derived unconfined failure strengths

Comparing the intercepts (fig 10b) shows that over the region where the stress range of the Brookfield PFT overlaps the other two testers, it shows agreement with the Jenike shear cell. The two Schulze RST cells show lower but consistent intercepts. At the two higher consolidation stress levels, the intercepts of the Schulze and Jenike cells cross over, with the former showing notably higher magnitudes. Into the low stress region the Brookfield PFT shows a strong fall off in the intercept magnitude as the consolidation stress approaches zero. If the Brookfield PFT data is interpreted in the same manner as the other two testers (see “PFT geometric method 2” in fig 10b) then the magnitude of the intercept is approximately midway between the Jenike and Schulze cell measurements.

The calculated unconfined failure strengths (the greater intercept of a Mohr passing through the origin and tangent to the assumed linear locus) are presented in fig 10c as a function of consolidation normal stress (not the principal stress). Inspection shows that the calculated unconfined failure strengths are reasonable consistent from all the different shear cells. Applying the same method of analysis to the Brookfield data as the other two cells “PFT geometric method 2” generates a slight reduction in unconfined failure strength, from 3.7 to 3.5kPa at a 4.8kPa consolidation normal stress.

6.1 Comparison of flow functions and effective friction functions

This section presents a comparison of the flow properties that have been derived from the failure loci, principally the flow function, but also the effective angle of internal friction function. The method used to determine these properties was described earlier in section 3.4. The mean flow functions and effective friction functions determined from round robin tests with the Brookfield PFT, Jenike shear cell and Schulze cell are presented in fig 11 & 12 respectively. Note that for the Brookfield tests, the standard deviations of the flow and friction functions were determined from scatter in the calculated values.

For the Jenike shear cell the mean data for the flow function (and internal friction) is that presented in the BCR report [7]. However error limits for these functions are not stated. To estimate these, the upper and lower error limits for the loci were used to determine the maximum and minimum variation in the consolidation stress σ_1, the unconfined failure strength σ_c and effective angle of friction δ_j. This was then used to construct the error bars shown in fig 11 & 12. For the case of the Schulze cell, the author was not aware of any published values for the mean flow properties or their standard deviations. Therefore the mean flow function and effective friction function were calculated from the published mean loci of [10] following the ASTM standard method [16]. The standard deviations were calculated using the approach outlined above for the Jenike data. For the error bars calculated for the Schulze data see figs 11.

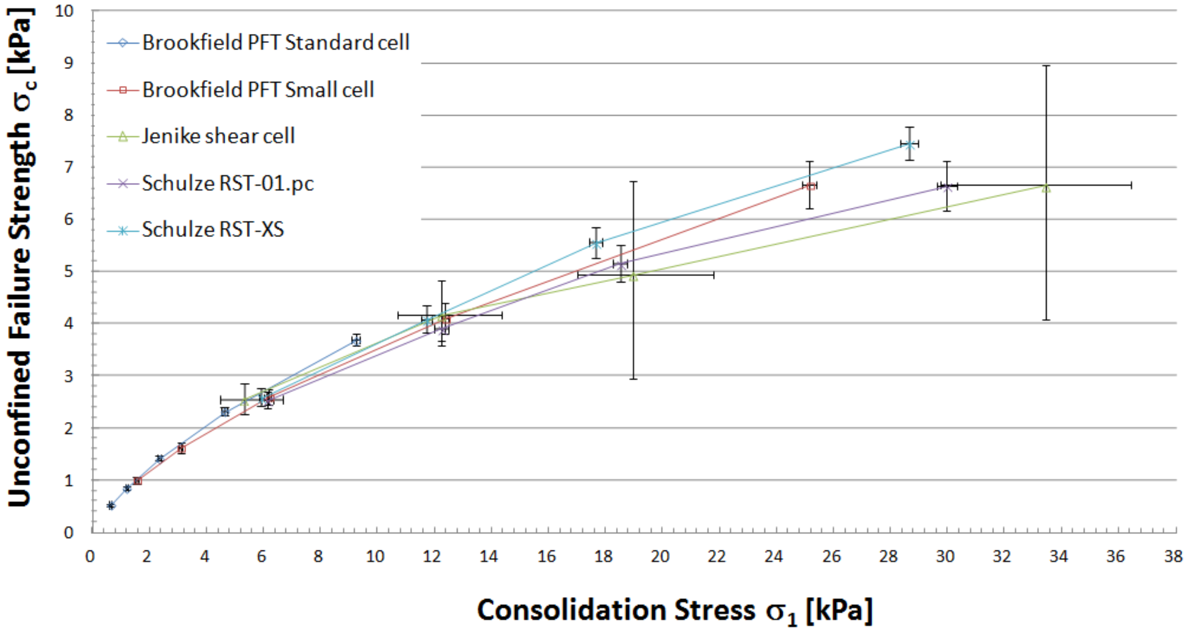


Fig 11, Comparison of the mean flow functions generated from round robin trials with BCR limestone (Note that flow functions and error bars for Jenike and Schulze cells were determined by the author using the technique described in the text of this article)

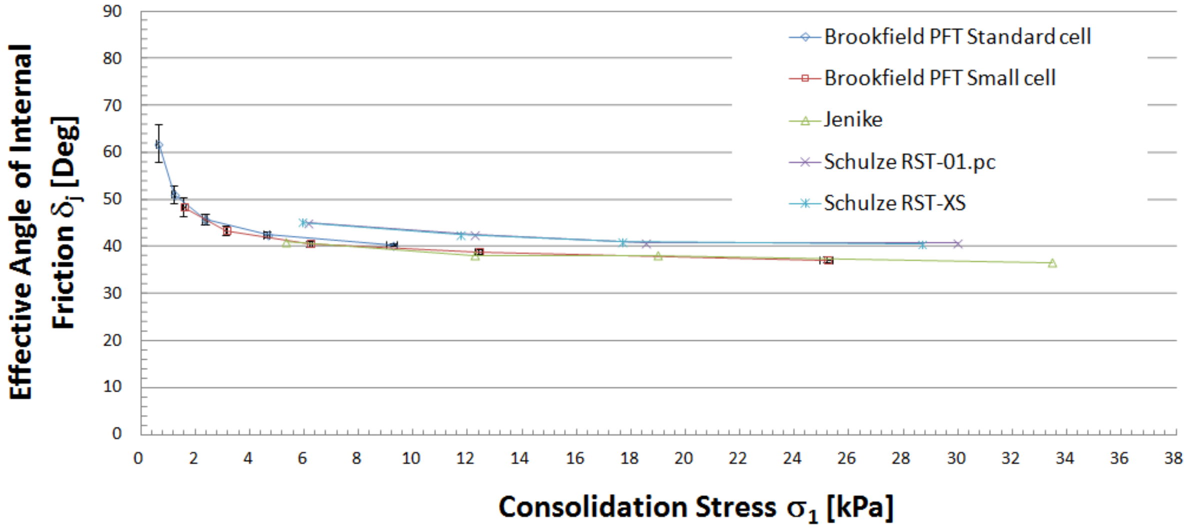


Fig 12, Comparison of the mean effective internal friction functions measured from round robin trials with BCR limestone in different shear testers

Inspection of fig 11 shows that mean values for the Jenike shear cell, Schulze RST's and Brookfield PFT give comparable measurements for failure strength (flow function) albeit with varying magnitudes of scatter in the repeat tests over the consolidation stress ranges. Figure 12 shows that the Schulze cells measure effective angles of friction that are approximately 4^0 higher than the Jenike and the Brookfield PFT shear cells.

Regarding the different cell sizes, the smaller Schulze RST-XS, measures higher strengths than the larger 01.pc, whereas the standard volume cell for the PFT measures higher strengths than the small volume cell.

7.0 Discussion

Broadly the comparison of the loci for the, flow functions and friction functions gives a few common observations.

The level of scatter is machine dependent, the Jenike giving significantly more scatter than the automated torsional testers. The Jenike cell also has a negative intercept for the loci of pre-shear points. Neither of these facts are a surprise due to the inherent difficulties in achieving critical consolidation in the Jenike cell [7] by comparison with the torsional testers. That is, the limited shear displacement (approximately 4mm), prevents the bulk solid sample from being sheared to critical state prior to failure. To determine critical consolidation the standard procedure for the Jenike shear cell requires a series of exploratory tests [1, 16 & 21] where the lid is subjected to a number of twists under a normal load in excess of the consolidation value. This process is repeated varying the number of twists and pre-consolidation load until the operator is satisfied that the procedure yields a critically consolidated sample. A new sample is then prepared for each failure measurement following the previously established consolidation technique. The above factors dictate that a lengthy testing time and extremely high level of operator skill are necessary to yield consistent results.

That said, the most significant difference in the Jenike cell results for the BCR standard is between the labs running the tests (of which there were 5) rather than reproducibility within individual labs. This is illustrated by fig 13 which is a duplicate of fig 12 except that the mean Jenike data has been replaced by 5 data series representing the mean flow functions measured by each Lab (A to E). Three of the Labs A, B, C show similar means, Lab D has a mean which is low across the stress range while Lab E is high at the 2 lower stresses, but very low at the 2 higher stresses. The data from Lab E is clearly in error with the 15 & 9kPa loci crossing over the 6kPa loci, resulting in an unconfined failure strength that reduces with increasing stress! It is the opinion of the author that this Lab E data (at least the 9 & 15kPa loci) should not have been included; its removal would increase the mean at the two higher consolidation levels and significantly reduce the level of scatter.

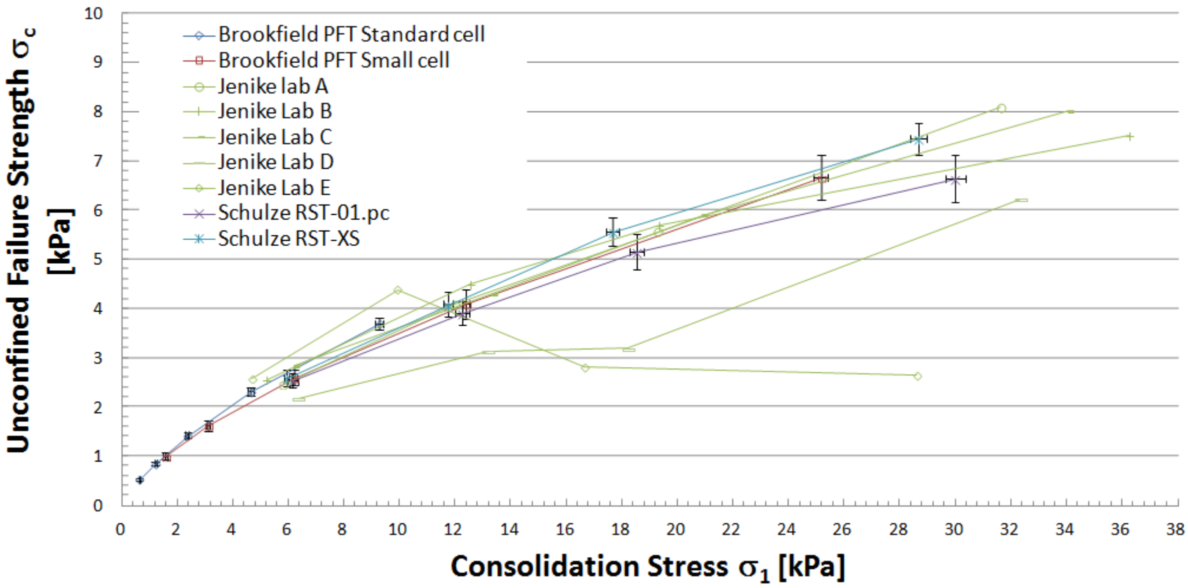


Fig 13, Comparison of the flow functions from the Jenike labs relative to other shear testers

Comparing the torsional tester failure loci and flow property results suggest that the:

- Brookfield PFT shows a slight reduction in scatter compared to the Schulze RST.

- The Schulze cells measure a higher shear stress at the consolidation end of the locus than the Brookfield PFT which is similar to the Jenike shear cell. As a result the Schulze cell measures a higher effective internal friction angle δ_j.

- The Schulze cell measures steeper failure loci than the Brookfield PFT which offsets the higher shear stress at the pre-shear point, resulting in lower intercepts but similar magnitudes for unconfined failure strength. This is shown clearly in fig 7 where loci have been measured at the same 3kPa normal stress and the average for the PFT was significantly lower but also displays a shallower gradient than that of the RST. When the loci are extrapolated back to zero normal stress the PFT data generates a greater cohesion, 0.78 versus 0.71 & 0.68 for the two RST cells. However, when comparing the unconfined failure strengths these show must closer agreement at 2.53, 2.55 and 2.6 for the PFT, RST 01.Pc & XS respectively.

- Thus the Schulze RST measures a slightly lower flow function than the Brookfield PFT, i,e. A similar unconfined failure strength but at a higher consolidation stress, for a given applied normal consolidation stress.

There are a number of possible causes for the discrepancies in the loci described above due to differences in the test algorithm and the shear cell geometry that effect the measurements. An investigation of the influence of the cell geometry, speed and test procedure on the results of torsional shear testers by Schmitt [22] gave the following ranking. The shear speed had no influence, the total shear strain (displacement) of the powder sample had a moderate influence and the shear cell geometry (design) had the most influence on the measured failure loci. The implications of these factors on the comparison between the PFT and RST are outlined below:

Regarding the shear speed the Brookfield PFT operates at shear speeds of 6.5 & 7.5mm/min at the mean radius of the standard cell and small cells respectively, whilst sampling torque at 100Hz. These are within the 1.5 to 7.5mm/min range over which Schmitt [22] found no influence for the Schulze RST XS and 01.PC.

Regarding the test algorithm there are three principal differences; the procedure used to consolidate the sample prior to failure locus measurement (ratcheting versus shearing), the total shear displacement at each consolidation stress level and the use of separate samples for each level. The work of Schmitt [22] suggested that the effect of the shear displacement was a moderate affect but the other two factors were not tested as they are not part of the ASTM standard method. The significance of the magnitude of the shear strain as proposed by Schulze [23] is that; as the failure zone transitions from pure shear to simple shear, the width of shear zone narrows, which leads to a more consistent but slightly lower strength and hence lower friction.

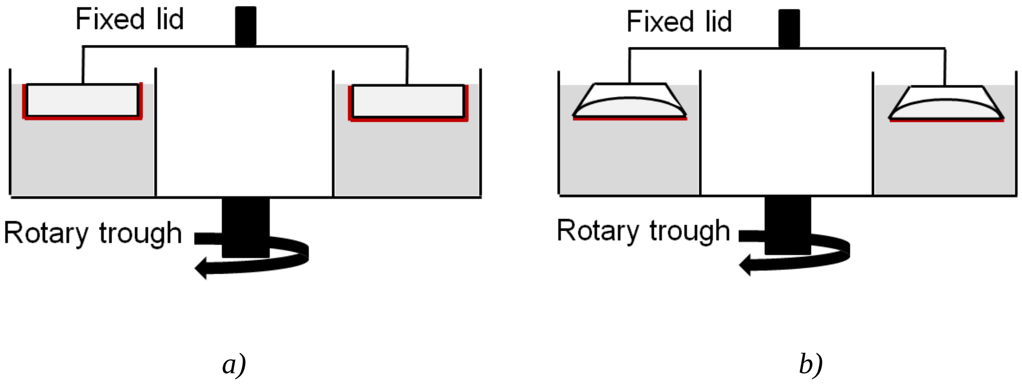
For the standard PFT on a single sample the shear strain required to consolidate and fail the sample, reduces as the consolidation level increases. Thus, at 0.3kPa consolidation normal stress level, all measurements are obtained within a shear displacement of approximately 21mm, reducing to 14mm shear displacement at the 4.8kPa consolidation normal stress level. The total shear displacement over all 5 stress level is of the order of 90mm. For the tests undertaken using a fresh sample at each consolidation stress level, the shear strain was found to be approximately constant at 21mm for all stress levels from 0.3 to 4.8kPa normal stress. Thus indicating that, the consolidation history of the sample reduced the shear displacement required for subsequent consolidation to the next stress level.

The shear strain used by the RST is not explicitly stated in the BCR report [10], however the work of Schmitt [22] reporting on titanium dioxide at a 5.5kPa normal stress level, found that RST procedure generated a shear strain of 17mm whereas the Peschl based procedure required 35mm. These differences were reported to give a moderate influence on the shear stress.

On the basis of the above information the shear displacement generated by the PFT using fresh samples for each consolidation stress are between the extremes reported by Schmitt for the Schulze RST and Peschl cell, but much closer to the former. When PFT test is run using a single sample for all stress levels the, shear strain at the 4.8kPa is significantly lower than that required by the Schulze RST procedure, but the powder has been subjected to shear at lower stress which has been shown to a very small effect on the flow function.

Thus the magnitude of strain at each consolidation level might be similar, but the RST is likely to have subjected the powder to a lower value. **Regarding the shear cell geometries**, the annular lids of the two cells are very different. The Schulze has open pockets (flat lid with vertical vanes) whilst the Brookfield uses closed pockets (radiused pockets) as shown respectively in fig14a&b. The results of tests undertaken by Schmitt [22] found that the standard Peschl cell which featured a flat lid with a knurled contact surface “to grip the powder” measured lower shear stresses than the same lid when fitted with “open pocket” vanes of the same dimensions used on the Schulze RST lid. Similar experiments on a manual annular shear tester at The Wolfson Centre during the development of the

PFT using both ‘open’ and ‘closed’ pocket lids found that the former measured a higher shear stress for a given normal stress [4] when following the same testing procedure. Tests also found that using a flat lid lined with a coarse sand paper gave similar results to the closed pocket lid, while switching to an open pocket lid gave an increase in shear stress, approximately proportionally to the vane depth. A possible explanation for this might be that, as the powder was consolidated under the major principal stress σ_1, lateral stresses were transferred in the intermediate principal stress direction σ_2 (from the open sides of the pocket) to the inner and outer circumferences of the trough wall. The presence of these stresses created additional frictional forces which were not present in the ‘closed pocket’ lid. Both differences in the shear algorithm and cell geometry above would be expected to result in the Schulze RST cell measuring a locus with a higher shear stress and effective internal friction.



a) *b)*

Fig 14 Schematic diagram of a cross section through the different types of lid pocket design; a) open, b) closed

8.0 Concluding Remarks

The Brookfield Powder Flow Tester represents the first shear tester that has been developed for economic measurement of powder flow properties for the purposes of formulation, purchasing and quality control, rather than silo design. However the tester broadly follows the measuring technique laid out in ASTM D6123 (as discussed in section 3.4) and can therefore be used for silo design. The Wolfson Centre has been successfully using the machine in this role for the last 3 years.

It is hoped that in time the machine will bring a level of understanding of powder flow to a large number of processing industries, thereby reducing the number of instances of powder flow problems.

The limitations of the machine are the minimum sample volume of 43cc which is large by comparison with other available machines (Schulze RST-XS and Freeman FT4 shear cell). The maximum consolidation stress σ_1 attainable in the standard volume cell 263cc is significantly lower (approx. 8-12kPa depending on level of shear stress) than afore mentioned machines. In terms of limitations to the powders/ particulates that can be tested, the maximum particle size for the standard cell is 2mm and it is unsuited (like any shear tester) to testing fibrous bulk solids. Regarding its applications to silo design, the main limitation is the maximum consolidation stress level. Using the small cell, the standard flow function test approximately covers the stress range dictated by the ASTM 46128 for bulk solids with bulk densities up to 800kg/m^3. For materials with bulk densities from 1800 to 2400kg/m^3 it covers half the stress range so an alternative machine would be required for the highest consolidation measurement, A direct comparison of the Brookfield PFT with the standard results of the Jenike and Schulze cells was not possible due to a disparity in the operating stress ranges of the machines. The high stresses used in the standard [7] are dictated by the inability of the Jenike shear tester to measure at low consolidation stresses [21]. Thus the lower stress range used for the Brookfield is actually more useful for quantifying flow problems in industrial processes or undertaking mass-flow silo outlet designs calculations [1] for instantaneous conditions.

Over the ranges of stresses where the measurements from the different machines overlap, the BCR limestone round robin tests have found the following. While there are small differences in the relative positions and gradients of the loci measured from the different testers; .i.e. the Schulze measuring a higher shear stress, the Jenike a lower shear stress, the PFT in between, the net effect on the flow function, the primary measurement is, small. Both the Brookfield PFT and Schulze RST give similar reproducibility. The Jenike shear cell data is order of magnitude more scattered and the mean significantly lower. However this is due to inconsistencies' of the data from two of the five labs participating in the trials.

The higher shear stress measured by the Schulze RST will result in a slightly higher internal friction angle than the Jenike and the PFT. Thus for silo design application this would give a less conservative design, a fractionally larger mass-flow hopper half angle and slightly smaller outlet dimension for a given angle of wall friction ϕ_w.

9.0 Acknowledgements

The development of the Brookfield PFT was funded by Defra through the Advanced Food Manufacturing Link Programme (AFM206) from 2004-2007 and by Brookfield Engineering Laboratories.

The shear tests with Brookfield PFT and limestone powder CRM 116 were funded by Brookfield Engineering Laboratories.

10.0 Nomenclature

c	cohesion intercept of linear Jenike failure locus	[kPa]
m	gradient of linear Jenike failure locus	
δ_j	Effective angle of internal friction	[deg]
ϕ_w	Effective angle of wall friction	[deg]
θ	Hopper half angle	[deg]
ρ_B	Bulk density	[kgm^{-3}]
σ_N	Normal stress	[kPa]
σ_1	Major principal consolidation stress	[kPa]
σ_2	Intermediate principal consolidation stress	[kPa]
σ_c	Unconfined failure strength	[kPa]
σ_{crit}	Approximate critical unconfined failure strength (arching or rat-holing)	[kPa]
σ_E	Consolidation normal stress	[kPa]
σ_w	Wall normal stress	[kPa]
τ	Shear stress	[kPa]
τ_E	Peak shear stress	[kPa]
τ_{ss}	Steady state shear stress	[kPa]

11.0 References

[1] A. W. Jenike, Gravity flow of bulk solids, Utah Univ. Eng. Exp. Stn. 123, 1964.

[2] R.J. Berry, M.S.A. Bradley, Investigation of the effect of test procedure factors on the failure loci and derived failure functions obtained from annular shear cells, Powder Technology, 174, (2007), 60-63.

[3] R.J. Berry, M.S.A. Bradley &K. Ariza, Interpretation Of Stick-Slip Powder Flow-Ability Measurements, 6th International Conference for Conveying and Handling of Particulate Solids (ChoPS-06 2009), Brisbane, Australia, August 2009.

[4] R.J. Berry & M.S.A. Bradley, An Investigation Of The Effect Of Annular Shear Cell Geometric Factors On The Measured Failure Loci And Derived Failure Functions, 5th International Conference for Conveying and Handling of Particulate Solids (ChoPS-05 2006), Hilton Hotel, Sorrento, Italy, Aug 27-31, 2006.

[5] R.J. Berry & M.S.A. Bradley, Comparisons Between Observed Powder Behaviour In Industrial Feeders And Measured Powder Failure Properties Obtained Using A Short Cut Silo Design Procedure, 5th International Conference for Conveying and Handling of Particulate Solids (ChoPS-05 2006), Hilton Hotel, Sorrento, Italy, Aug 27-31, 2006.

[6] R.J. Berry, M.S.A. Bradley, & R.J. McGregor, Development and commercialisation of a new Powder Flow Tester for powder formulation, quality control and equipment design, Bulk Solids India 2010, March 2010

[7] R. J. Akers, The certification of a limestone powder for Jenike shear testing CRM-116, Loughborough Univ. of Technology, UK, BCR/163/90 (Community Bureau of reference, 1990)

[8] D. Schulze, Flowability and time consolidation measurements using a ring shear tester, Powder handling & processing, Vol. 8, No. 3, July/September 1996, pp221-226.

[9] Freeman FT4 Powder Rheometer, Freeman Technology (UK).

[10] D. Schulze, Round Robin Project - Results, Shear tests on limestone powder CRM-116 with Ring Shear Testers RST-XS and RST-01.pc (2009), available as pdf at *http://www.dietmar-schulze.de/roundrobin.html*

[11] J. Cooke, T. Freeman, Validation of shear testing using the FT4 Powder Rheometer and Limestone Powder CRM 116

[12] I.A.S.Z. Peschl, Equipment for the measurement of mechanical properties of bulk materials, Powder handling and processing, Vol. 1, No. 1, March 1989, pp 73-81.

[13] J.R. Johanson, The Johanson Indicizer^{TM} System vs. the Jenike shear tester (New technology simplifies solids flow properties characterization), Bulks solids handling, Vol. 12, No. 2, May 1992, pp237-240.

[14] T. A. Bell, B. J. Ennis and R. J. Gryco, USA, Scholten W. J. F. and Schenkel M. M., The Netherlands, Practical evaluation of the Johanson Hang-up Indiczer, Bulk Solids Handling, Vol. 14, No. 1, Jan/Mar (1994), pp.,

[15] G.G. Enstad & L.P. Maltby, Flow property testing of particulate solids, Bulk solids handling, Vol. 12, No. 3, Sept. 1992

[16] ASTM Designation: D 6128-06, Standard Test Method for Shear Testing of Bulk Solids Using the Jenike Shear Cell (2009)

[17] J. F. Carr and D. M. Walker, An Annular Shear Cell for Granular Materials, Powder Technology, Vol. 1, (1967/68), pp., 369-373.

[18] I.A.S.Z. Peschl, Mechanical Properties of powders, bulk solids handling, Vol. 8, No. 5, (Oct 1988) 615-624.

[19] I.A.S.Z. Peschl, Principals of soil mechanics for the characterisation of industrial powders, Powder Handling & Processing, Vol. 13, No. 1,(Jan/Mar 2001) 11-18.

[20] D. Schulze Powders and Bulk Solids, Behaviour, Characterisation, Storage and Flow, Springer, 2007.

[21] J. Schwedes, Review on testers for measuring flow properties of bulk solids, Granular Matter 5, 1-43, 2003

[22] R. Schmitt, H Feise, Influence of Tester Geometry, Speed and Procedure on the Results from a Ring Shear Tester, Part. Part. Syst. Charact. 21 (2004) 403-410.

[23] D. Schulze, H. Heinrici, H. Zetzener, Powder Handling and Processing 2001, 13, 19.