Limestone Powders Yielding and Steady State Resistance under Shearing with Different Testers

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ABSTRACT We study the effect of both particle size and shear testers on the failure (yielding) and the steady state shear strength of granular materials. Physical experiments are carried out on four fine limestone powders using a geotechnical direct shear tester and the standard Schulze ring shear tester to explore both material behaviors and device influence. The direct shear tester is validated by comparing the yield locus measurements with the Schulze ring shear tester. A good agreement is found between these two testers and this unveils the possibility of using different testers across different scientific communities/regimes. The termination locus is then studied in direct shear tester for the four limestone powders. A pronounced particle size effect is evident on both the angle of internal friction and the steady state angle of internal friction, which highlight the effect of the inter-particle cohesion.

1. INTRODUCTION

Granular materials are ubiquitous in our daily life. A special class is powders, which contain fine particles that may flow freely when shaken or tilted enough, but on the other hand, the particles can stick together to form aggregates/agglomerates. Particles smaller than 100 μm are usually found to be cohesive [1]. The likelihood of cohesion increases with decrease in particle size. During storage and transportation in the industry, the powder which consists of many independent particles faces various stress conditions, such as compression, abrasion, shearing, etc. The powder may show peculiar flow behavior such as segregation, clustering, shear-band formation, and arching, etc. [2]. A topic of particular relevance from the application point of view is to understand the yield behavior of powders, *i.e.*, when they start flowing under shear, and related question that what is the shear stress necessary to keep them flowing. In powder technology, yield loci are obtained generally from shear tests. The most commonly used testers are the Jenike shear cell (a shear cell with circular cross-section) [3] and the Schulze ring shear tester [4, 5]. The latter has the advantage compared to Jenike that it has no shear displacement limit. In soil mechanics, the direct shear box (with square cross-section) is the most commonly used device to obtain yield loci for clay and sandy soils, as introduced by Casagrande [6, 7]. This device has the possibility of reversal displacement for cyclic loading testing. Moreover, this device also allows to measure the termination locus easily (in the steady state after long shear) [8].

In order to understand how particle size influences the bulk characteristics of powders and to test the reproducibility of measurements in different shear devices, we study the yield locus of four different limestone powders with different median particle size ranging from 2.2 to 138 μm in both direct and Schulze tester. Then we further measure the termination locus using the direct shear tester (because of having less flexibility when using the highly automated Schulze ring shear tester). In section 2 the focus is on material description. The experimental set-up and methods are presented in section 3, while results and discussion are presented in section 4. Finally, conclusions with outlooks are given in section 5 with an outlook.

2. MATERIAL DESCRIPTION AND CHARACTERIAZATION

The Eskal® series of calcium carbonate/limestone powders are commercially available from KSL Staubtechnik GmbH, Germany and are used extensively in many applications in construction, food and oil industries. They are also used as a standard reference testing powder for the calibration of equipment in powder technology, including shear testers [9, 10] and optical sizing systems due to some favourable physical properties. They are nearly spherical in shape, non-porous and insensitive to humidity and temperature changes, which allows them to be used without change in their behaviour. Therefore, Eskal® series has been selected as reference powder and used for two collaborative European projects, see: www.pardem.eu [11, 12] and www.t-mappp.eu [13].

In Table 1, we summarize the physical properties of the limestone powders used for this study. Our median particle sizes range from 2.22 μm (cohesive, primary particles stick to each other and form clumps) to 138 μm (free-flowing, primary particles stay separate from each other) were measured using the laser diffraction (HELOS) method. The span of the particle size distributions decreases with increasing particle size from 1.52 to 0.7 while the initial bulk density (initial sample state after filling) increases from 540 to 1370 kg/m^3. The particle density is measured using a Helium pycnometer. All materials have the same particle density and the moisture content.

Table 1 Material parameters of the experimental samples. The initial bulk density represents bulk density from raw materials.

Properties	**Eskal300**	**Eskal500**	**Eskal15**	**Eskal150**
Particle Density ϱ_p [kg/m^3]	2737	2737	2737	2737
Median Particle Size d_{50} [μm]	2.22	4.42	19	138
Span [-]	1.52	1.50	0.84	0.70
Moisture Content ψ [%]	0.9	0.9	0.9	0.9
Initial Bulk Density ϱ_{b0} [kg/m^3]	540	730	1110	1370

Figure 1 shows the scanning electron microscopy image of Eskal15 at low and high magnification. The images are analysed using the backscattering electron method to detect contrast between areas with different chemical compositions. Backscattered electrons (BSE) consist of high-energy electrons originating in the electron beam, that are reflected or back-scattered out of the specimen with different intensities, depending on the chemical compositions at their point of impact. We observe similar brightness of primary particles in both large and small scale, which means our limestone samples have almost uniform chemical composition. When looking at the other Eskal powders, they all have more or less similar shape and chemical composition irrespective of their median particle size (data not shown here).

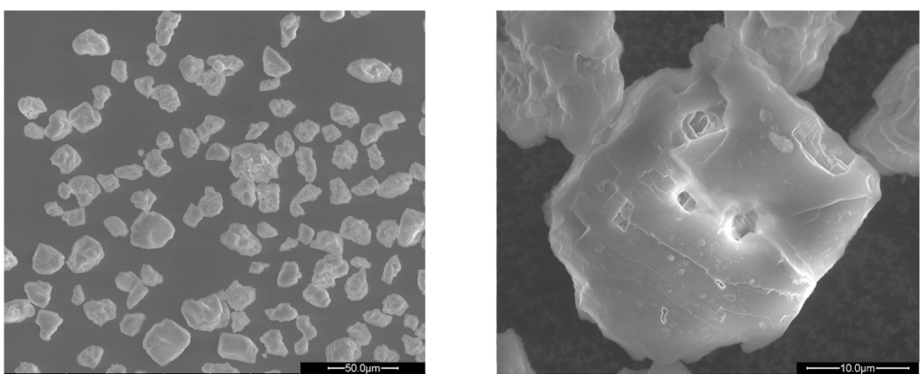

Figure 1 Scanning Electron Microscopy (SEM) images of Eskal15 using backscattered electron method for the composition of primary particles in low (left) and high (right) magnification, with the scale given at bottom right.

3. EXPERIMENTAL SET-UP AND TEST DETAILS

Two experimental shear testers are used in this work: the direct shear tester (ELE International, UK), illustrated in Figure 2 and the Schulze ring shear tester (RST-01), as shown in Figure 3. For sake of brevity, we will refer to the direct shear tester as DST and to the ring shear tester as RST in the following.

The DST accepts specimens with 60 mm in length and width and 30 mm in height. The apparatus is enclosed in a robustly constructed case since it has been designed for floor mounting and it is supplied with carriage, loading hanger and 10:1 lever loading device, which allows shear stress up to 1.25 MPa and normal stress up to 2.78 MPa. The speed range of shear is between 0.0001 to 2 mm/min and our shear rate is chosen as fastest 2 mm/min fror all the tests.

Figure 2 The ELE direct shear tester (left) and the schematic representation of the shear cell set-up (right).

The advantages of the DST are a high load design for soil samples and a simple shear principle similar to the Jenike shear tester, but with a larger shear path (up to 10 mm in horizontal direction) and the possibility of reversing strain path. In order to enhance the reproducibility of yield locus measurement, the standard test procedure from the Schulze shear tester is ameliorated for the direct shear tester.

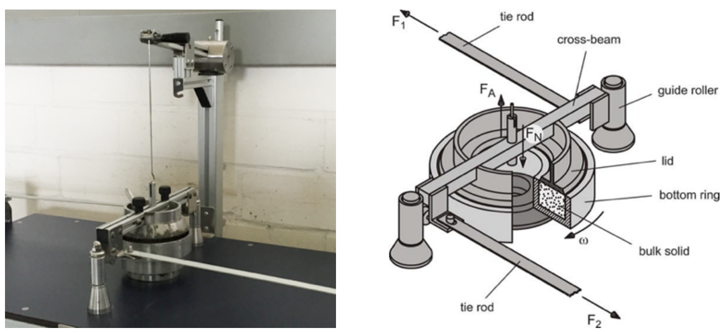

Figure 3 The Schulze ring shear tester (left) and the working principle of the shear cell set-up (right) [4].

The RST is a shear tester designed based on the working principle of the Jenike shear cell. It accepts a ring shape specimen of height 24 mm, inner/outer diameter 60/120 mm. The maximum normal stress is 50 kPa, with shearing speed between 0.002 and 18 mm/min and shear speed used is same as for DST. The RST is highly automated and has the advantage that there is an infinite shear path available to get the specimen through pre-shear into steady state. The standard ASTM-D6773 [14] yield locus measurement procedure is used for both shear testers. Firstly, we apply a given normal stress (36.1 and 35.0 kPa for DST and RST respectively) and pre-shear until the shear stress saturates at that given normal stress, then we stop shearing and reduce the shear stress to zero (pre-shear stage). After that, the normal stress is brought to a certain lower value and the specimen is sheared until failure is reached (shear stage). This loop (pre-shear + shear stages) is repeated five times, by using a unique normal stress value for pre-shear and five different values of normal stress in shear. Thus, a five shear points yield locus is obtained. A slightly different procedure is used to measure the termination locus: we step-wise increase the normal stress and shear the specimen until the shear stress reaches the steady state for each normal stress level. The detailed test procedures are explained in [4, 13]. The stress levels used in this study for the yield locus are summarized in Table 2 and in Table 3 for termination locus. All the tests are repeated 3 times in order to get statistics and reproducibility of testers.

Table 2 Yield locus test details on four limestone powders. Note that pre-shear and shear refer to the normal stress applied for shearing until steady state and peak failure state respectively.

Device	Sample	Normal Stress Applied	Value (kPa)	Repetitions
DST	Eskal 300, 500, 15 and 150	Pre-shear	36.1	3
		Shear	1.4 to 30.5	3
RST	Eskal 300, 500 ,15 and 150	Pre-shear	35.0	3
		Shear	2.0 to 20.0	3

Table 3 Termination locus test details on four limestone powders.

Device	Sample	Normal Stress Applied	Value (kPa)	Repetitions
DST	Eskal 300, 500, 15 and 150	Pre-shear	1.4 to 36.1	3

4. RESULTS AND DISCUSSION

In this section, we will first compare the yield locus measurements from two shear testers for two limestone powders, then we study the termination locus measurements using DST. Finally, the influence of particle size and shear testers on the bulk properties is addressed.

In Figure 4, we plot the results for Eskal15 and 500 using both shear devices. If we look at the loci from DST, they show linear behavior with small standard deviations (1.5-12%). For the yield loci measured in the RST, a better self-reproducibility is obtained with even smaller standard deviations (0.8-4.5%). The yield loci measured from DST lay in between the results from RST with reasonable differences (1.7% and 7.0% for Eskal15 and Eskal500 respectively). This means that there is no device dependence. A similar behavior is found when testing the other two powders, Eskal150 and 300 (data not shown here), using the same testers. Therefore, we confirm a quite good agreement between DST and RST in the stress range we have investigated.

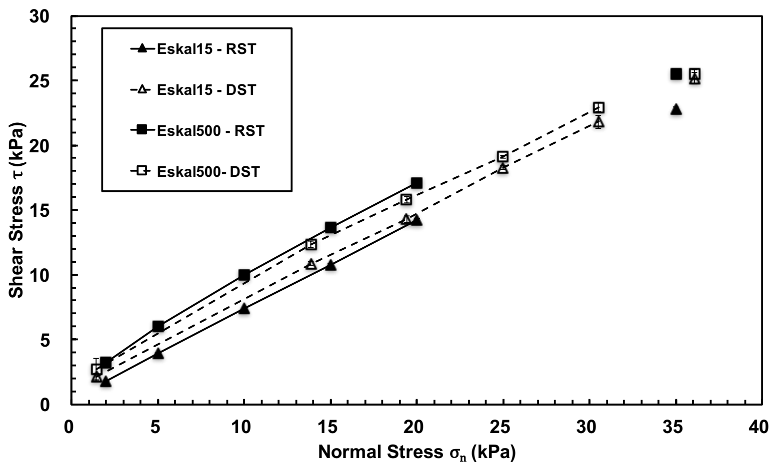

Figure 4 Yield loci of Eskal15 and 500 using the ring shear tester (RST, with solid lines) and the direct shear tester (DST, with dotted lines), the points without lines on the right are the pre-shear/steady-state points. Note that here the lines are only guides to the eye.

After the reproducibility of the behavior between the two shear testers is established, we further test the termination locus using DST and results are shown in Figure 5. Note that here all points are in the same steady state as pre-shear points shown in Figure 4.

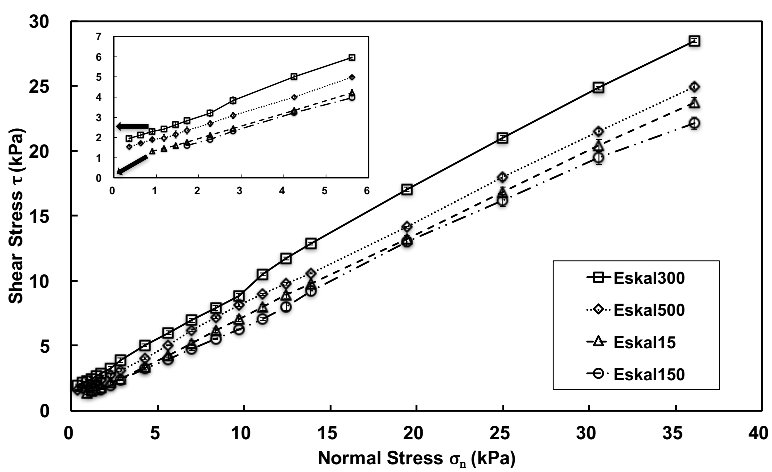

Figure 5 Termination locus measured for four limestone powders using direct shear tester (DST). The inset is the enlargement into the low normal stress regime (0 to 6 kPa). Lines are only guides to the eye.

For Eskal 500 and 300, the slopes of the loci change at some stress levels (3 and 11 kPa) indicating that cohesion is enhancing the shear resistance of the specimen. When we look at the low normal stress regime, as given in the inset, the curves for Eskal300 and 500 are tending towards a rather large finite values of about 1.8 kPa and 1.3 kPa. We have no clear offset visible for Eskal15 and 150 due to the limited reliable data at very low stress, below 1 kPa. It is still questionable whether the termination locus will tend towards the origin or has a finite intercept value (two arrows shown in the inset), both being possible given in our data.

In order to explore the also influence from particle size, the angle of internal friction (slope of a linear fit to the yield locus) and the cohesive strength (interception of the extrapolated linearized yield locus to zero normal stress) are extracted from the fitted yield loci. In Figure 6 (left), we show the relationship between the angle of internal friction and the median particle size of the four limestone powders in the two testers. We get a decreasing trend with increasing particle size in both DST and RST, with the DST values consistently 1°-3° lower than the RST values and much higher standard deviation (1-5%) compared to the RST (0.4-1%). In the same plot, the steady state angle of internal friction has the same decreasing trend with increasing particle size. The values are consistently higher than the angle of internal friction measured from yield locus by about 1 degree. This is expected and consistent with previous results. The cohesive strength both extrapolated from yield locus and termination locus are shown in Figure 6 (right). The value of cohesive strength measured by the RST approaches zero with increasing particle size, but the values from the DST saturate at around 1 kPa for particle sizes larger than 10 μm. This 1 kPa value is also the lower limit of the DST force sensor, around which we get really high standard deviations. For the sake of completeness, we report the cohesive strength in the steady state here in the same figure, however, these data are not reliable due to the extrapolation of high deviations in low normal stress end.

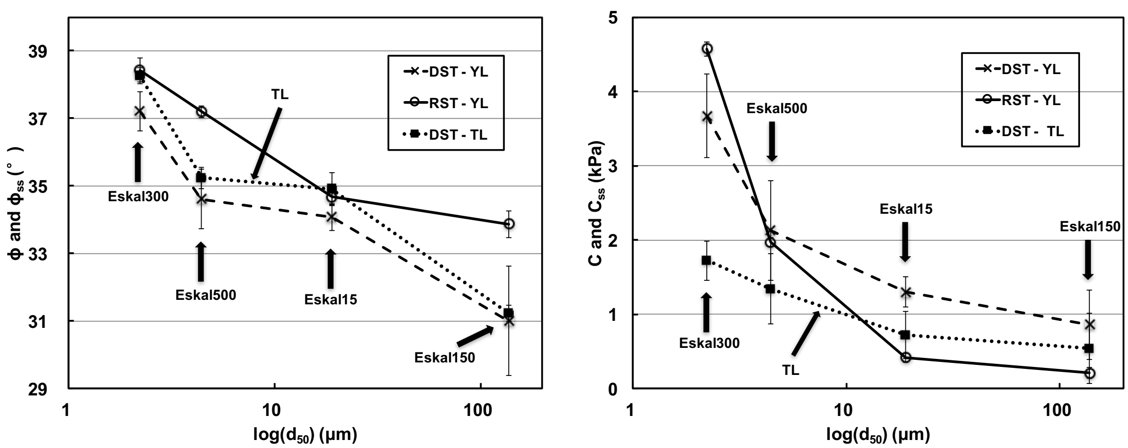

Figure 6 Left: Angle of internal friction measured from yield locus (YL), ϕ, and from the termination locus (TL), ϕ_{ss}; Right: cohesive strength measured from the yield locus extrapolation to zero normal stress, C, and from the steady state, C_{ss}, plotted against the median particle size, d_{50}, in semi-log scale using data from both direct shear tester (DST) and ring shear tester (RST). The values are obtained by linearized fitting of all the loci presented before.

5. CONCLUSIONS AND OUTLOOK

Two commonly used shear testers (ELE direct shear tester DST and Schulze ring shear tester RST) have been used, two different shear states (yield locus and termination locus) are studied for dry limestone powder with median particle sizes ranging from 2 to 138 μm. Different cohesion is observed with similar chemical properties. Despite a higher standard deviation for the measurements in the DST, good agreement is obtained between the two testers for all the powders and stresses presented in this study.

The particle size is one major influencing factor for the bulk behavior. The cohesive strength, the bulk friction in the failure state (angle of internal friction) and the steady state (steady state angle of internal friction) all decrease with increasing particle size.

For future work, experiments with a wider range of particle sizes (and thus different cohesion) is planned to confirm the decreasing trends and correlations with size. Further comparison with other, also newly developed shear testers is in progress. The link between both bulk friction and cohesion experimentally and numerically (using the Discrete Element Method) will also be addressed.

6. ACKNOWLEDGEMENT

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7. NOMENCLATURE

ϱ_p	: particle density [kg/m^3]
ϱ_b	: bulk density [kg/m^3]
ϱ_{b0}	: loose poured initial bulk density [kg/m^3]
d_{50}	: median particle size [μm]
σ_n	: normal confining stress [kPa]
τ	: shear stress [kPa]
τ_p	: peak failure shear stress [kPa]
τ_{ss}	: steady state shear stress [kPa]
ϕ	: angle of internal friction (yield locus) [°]
ϕ_{ss}	: steady state angle of internal friction (termination locus) [°]
C	: cohesive strength of yield locus [kPa]
C_{ss}	: cohesive strength of termination locus [kPa]
ψ	: moisture content [%]

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