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Slow stress relaxation behavior of cohesive powders

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ABSTRACT

We present uniaxial (oedometric) compression tests on two cohesive industrially relevant granular materials (cocoa and limestone powder). A comprehensive set of experiments is performed using two devices – the FT4 Powder Rheometer and the custom made lambdameter – in order to investigate the dependence of the powders' behavior on the measurement cell geometries, stress level, relaxation time and applied strain rate. The aspect ratio α, tested with the FT4, is found to play no role for vessels with α ≤ 1 while material characteristics strongly affect the stress–strain response. After compression is stopped, the constant volume stress relaxation is found to follow a power law, consistently for both cohesive powders and for the different testing equipments. A simple (incremental, algebraic) stress evolution model is proposed to describe the relaxation of cohesive powders, which includes a response timescale along with a second, dimensionless relaxation parameter that sets the very small power law, i.e. extremely slow stress relaxation.

1. Introduction and background

Granular materials are omnipresent in nature and widely used in various industries ranging from food, pharmaceutical, agriculture and mining — among others. In many granular systems interesting phenomena like dilatancy, anisotropy, shear-band localization [49], history-dependence [52], jamming and yield have attracted significant scientific interest over the past decade [1,19,32]. The bulk behavior of these materials depends on their constituents (particles) interacting through contact forces [42,50]. To understand their deformation behavior, various laboratory element tests can be performed [47,35]. Element tests are (ideally homogeneous) macroscopic tests in which one can control the stress and/or strain path. Such macroscopic experiments are important ingredients in developing and calibrating constitutive relations and they complement numerical investigations [32]. Element test experiments on packings of bulk solids have been studied experimentally in the biaxial box [37,40,36] along with uniaxial compression and volume conserving shear [43], [58,38]. A detailed review of different testers has been presented in [47]. Additionally, element tests carried out with more complex, non-commercial testers have been reported in the literature [15,23,17,5], even though their utilizations are restricted to specific applications, e.g., to the testing of geophysically relevant materials at relatively high consolidation stress.

The testing and characterization of dry, non-sticky powders are well established. For example, rotating drum experiments to determine the dynamic angle of repose have been studied extensively as a means to characterize non-cohesive powders [39,6,8], even though these tests are not well defined with respect to the powder stress and strain conditions. The main challenge comes when the powders are sticky, cohesive and less flowable like those relevant in the food industry [21]. For these powders, dynamic tests at low stress, like in a rotating drum, are difficult to perform due to contact adhesion and clump formation. In order to overcome this problem, it is preferable to perform confined tests at higher consolidation stresses, where the stress and strain paths can be well defined and better controlled, so that reproducibility is enhanced.

One element test which can easily be realized (experimentally and numerically) is the uniaxial (or oedometric) compression (in a cylindrical or box geometry) involving deformation of a bulk sample in the axial direction, while the lateral boundaries of the system are fixed. This test is particularly suited for determining the poroelastic properties of granular materials [18,19,20,4]. While most uniaxial tests on dry bulk solids have been devoted to studying the relationship between pressure and density and the bulk long time consolidation behavior, the dynamics of the time-dependent phenomena have been often neglected in experimental and practical applications [59]. For

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example, in standard shear testers like the Jenike shear tester [26] and the Schulze ring shear tester [45], during yield stress measurements, the focus is usually not on the relaxation behavior. Considerable stress-relaxation of bulk materials can even disturb yield stress measurements.

The stress relaxation phenomenon is similar to those observed in viscoelastic materials such as polymers [13,14,34], gels [9,57], in dielectric relaxation [24,25] and in the attenuation of seismic waves [27]. For viscoelastic materials, relaxation implies a memory effect and can be described using convolution integrals transformed to their fractional form and containing a relaxation modulus that describes the response of the system to stress [44]. For these materials, phenomenological models involving the combination of springs and dashpots, such as the Maxwell, Zener, anti-Zener, Kelvin -Voigt, and the Poynting -Thomson models have been developed (see Refs. [2,3,28,33] and references therein). Even though stress relaxation has also been observed in granular media [4,41,45,59], not much work has been done in providing a theoretical description of this phenomenon. Additionally, most cohesive contact models [32,53,54,56] used in discrete element simulation [21] of granular materials do not account for the time dependent relaxation behavior. For the improvement of both discrete element contact models and constitutive macromodels for cohesive powders, it is necessary to have an experimental and theoretical understanding of the long-term stress response of cohesive materials.

In the present study, using two simple devices, we perform oedometric compression tests with the main goal of investigating the relaxation of industrial powders at different stress levels. Another goal is to provide a quantitative comparison between the relaxation behavior as observed in two testers, namely the lambdameter [30,31,29] and the FT4 Powder Rheometer [12], in order to confirm that relaxation occurs irrespective of the device used. The lambdameter has the peculiar advantage that both vertical and horizontal stresses can be obtained simultaneously — unlike the FT4 Powder Rheometer and other simpler oedometric setups. Finally, we will propose a simple model that captures the stress relaxation of cohesive powders at different compaction levels.

The work is structured as follows: in Section 2, we provide a characterization of the material sample, and in Section 3 the description of the experimental devices and the test protocols. In Section 4, we present the theoretical model for stress relaxation. Section 5 is devoted to the discussion of experimental and theoretical results, while the conclusions and outlook are presented in Section 6.

2. Sample description and material characterization

In this section, we provide a brief description of the experimental samples along with their material properties. In order to investigate the relaxation behavior, two cohesive reference samples were chosen, namely cocoa powder and Eskal 500 limestone. The choice is based on several selection factors, among which are the suitability for different industrial applications, ability to withstand repeated loading without changes in the property and long term availability/storage of the samples.

The Eskal limestone has been used extensively as reference cohesive powder, and is made available in convenient amounts in a collaborative European project, c.f. www.pardem.eu [51]. Scanning Electron Microscope (SEM) images obtained using a Hitachi TM 1000 Instrument (Hitachi Ltd., Japan) for both powders are displayed in Fig. 1 and properties are displayed in Table 1.

The particle size distributions are measured using the Helos testing instrument (Sympatec GmbH, Germany). While limestone powder is dispersed with air pressure, we use the wet mode to disperse cocoa powder since it forms agglomerates. For the wet mode, cocoa powder is dispersed in dodecane, an oily liquid, in order to retain the fat layer while ultrasound (vibration) is applied to stress the dispersion and break off the agglomerates. The particle density is measured by helium

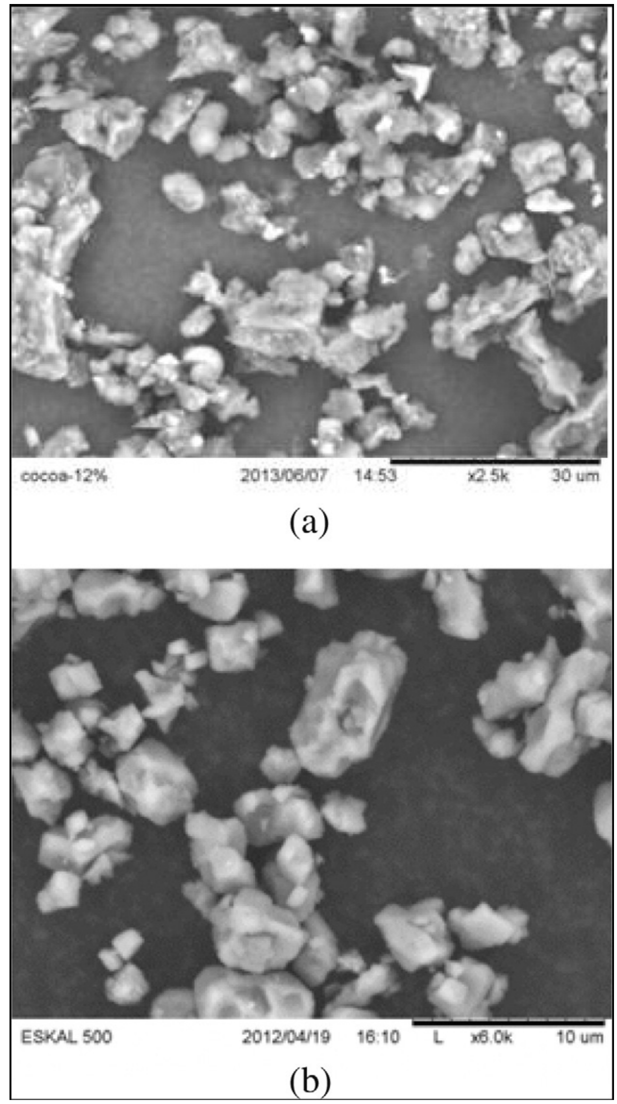

Fig. 1. Scanning electron microscope images of the cohesive samples (a) cocoa powder with 12% fat content (b) Eskal 500 limestone powder. Note the different scales at the bottom right.

pycnometry (Accupyc, Micromeritics, USA) while the moisture content is given as the ratio of the difference between the original and dried mass (after 24 h in an oven at 100 °C) and the original sample mass. The bulk cohesion is the limiting (extrapolated) value of shear stress for which the normal stress is equal to zero and is determined from

Table 1
Material parameters of the experimental samples. The letters L (Lambdameter), F (FT4 with $\alpha = 0.87$) and f (FT4 with $\alpha = 0.40$) indicate the initial bulk densities for the respective samples and setups.

Property		Unit	Cocoa (12%)	Eskal 500 limestone
Size distribution	D_{10}	μm	2.14	1.34
	D_{50}	μm	9.01	4.37
	D_{90}	μm	37.40	8.24
Particle density	ρ_p	kg/m^3	1509	2710
Moisture content	w	%	5.68%	0.25%
Bulk cohesion	σ_c	kPa	1.8 at 7.4 kPa	1.3 at 4.6 kPa
			9.6 at 41.8 kPa	3.3 at 12.7 kPa
Wall friction (lambdameter)	μ_w	[−]	0.25	0.58
Initial bulk density	ρ_0	kg/m^3	388 (L)	717 (L)
			450 (F), 467 (f)	855 (f)