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Cohesion of lactose powders at low consolidation stresses

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ABSTRACT

The flow characteristics of a powder system are known to be influenced by particle size distribution, particularly the content of fine particles, and interparticle forces. This paper reports an investigation that has identified and quantified links between physical properties, *viz* size distribution, bulk density and particle density, and cohesion in compacted beds of powder. An annular shear cell was used in the determination of the cohesion of cohesive and free-flowing milled lactose powders at low consolidation stresses in the range 0.31–4.85 kPa and under ambient conditions. Following consideration of the compaction and shearing processes, it was postulated and confirmed that cohesion could be expressed as a function of powder surface area per unit volume and dimensionless preconsolidation stress. It was shown that care is needed in the measurement of surface–volume mean diameter when applying correlations developed from the experimental data.

1. Introduction

Fine powders do not flow well due to their cohesive nature. Cohesion arises from interparticle forces that can exist in different forms, such as van der Waals forces, electrostatic or magnetic forces, mechanical interlocking between particles, capillary interactions, liquid bridging between particles, and combinations of these [1]. Cohesion is directly related to material physical properties such as particle size and size distribution, and poses significant influences on the mechanical and flow properties at the bulk level.

Powder cohesion can be determined using measurements made with shear cell apparatus, which is regarded as a standard for powder flow characterization [2]. The shear testing method was proposed by Jenike [3] and variants of shear cells have been developed over the years [2,4]. Shear testing at different preconsolidation stresses generates information on failure or yield loci. A yield locus is the relationship between shear stress, τ, and normal stress, σ, which has been reported to exhibit a concave curvature, see for example [2,5]. Extrapolation of the yield locus to the ordinate gives a value of powder cohesion. Cohesion in this context is the shear force required to shear a preconsolidated powder that has no applied normal stress [6]; it is a measure of internal forces within a powder system and depends on compaction history.

In this paper, the cohesion data for fine model lactose powders measured at low consolidation stresses with an annular shear cell are reported and modeled. The work forms part of an experimental campaign aimed at characterizing powder flow and providing information that can potentially benefit the food and pharmaceutical industries in which fine powders are important commodities. The emphasis is on the identification and quantification of links between powder cohesion, size distribution and consolidation stress.

2. Materials and methods

2.1. Materials

A total of 13 model milled lactose powders was used; each powder is identified by a code, as shown in Table 1. Three samples were used as received; they were lactose monohydrate Pharmatose® 70M (LP1) and Pharmatose® 350M (LP4), and milled Hydrous Refined Lactose 100-mesh (LM1), all commercial products of DMV-Fonterra Excipients, New Zealand. The other 10 samples were made by sieving either Pharmatose® 70M or the Hydrous Refined Lactose with BS 410 screens and an electromagnetic shaker (EMS-8, Mumbai, India). Two sieving procedures were used. In Procedure 1, 500 g of powder were sieved at 20 W for 20 min with selected British Standard sieves; lactose LM2, LM3, LM6, LM7, LM8, LP2 and LP3 were made following this procedure. Lactose LM4, LM5 and LM9 were made using Procedure 2 in which 50 g of powder were sieved at 20 W for 5 min.

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Nomenclature

A_p	surface area of powder (m^2)
C	powder cohesion (Pa)
d_{32}	surface–volume mean diameter (m)
$d_{32,M}$	surface–volume mean diameter measured with Mastersizer (m)
d^*_{32}	adjusted surface–volume mean diameter, sieve analysis equivalent (m)
$d_{50,M}$	particle size at 50% in a cumulative size distribution (m)
V_B	volume of bulk powder (m^3)
V_p	volume of particles (m^3)
a, b, m	regression parameters (units according to usage)

Greek letters

μ	coefficient of friction (–)
ρ_B	bulk density (kg m^{-3})
ρ_p	particle density (kg m^{-3})
σ	normal applied stress (Pa)
σ_c	major consolidation stress (Pa)
σ_D	major stress developed in a dome or pipe (Pa)
σ_{pre}	preconsolidation stress (Pa)
$\sigma_{pre,min}$	minimum preconsolidation stress (Pa)
σ_y	unconfined yield stress (Pa)
τ	shear stress (Pa)

2.2. Particle size

Particle size distribution was measured on the volume-weighted basis by the laser diffraction method (Mastersizer 2000, Malvern Instruments Ltd., UK). The equipment used the small volume sample unit with isopropanol as the dispersant and the 300 RF lens; the refractive index of lactose (1.533) and isopropanol (1.378), and the default *Polydisperse* model were used. The $d_{50,M}$ and $d_{32,M}$ for each material are given in Table 1; diameter $d_{50,M}$ is the particle size at 50% in a cumulative size distribution and $d_{32,M}$ is the surface–volume mean diameter measured with the Mastersizer. The model lactose powders have been listed in order of their $d_{32,M}$ to assist in identifying trends related to particle size.

2.3. Cohesion and powder flow functions

Shear tests were done with an annular shear cell (Brookfield Engineering Laboratories Inc., USA) under ambient conditions (20–24 °C, 36–54% RH). The shear cell was connected to a computer and controlled online with customized software. The two test options selected were *Geometric Spacing of Consolidation Levels* and *Measurements at the Tangent Load*. A standard procedure was used for each sample: preconsolidation and shearing to a critical state, followed by shearing at a lower normal stress to obtain a yield point. A family of yield loci was created for five preconsolidation stresses, σ_{pre}: 0.31 kPa, 0.61 kPa, 1.20 kPa, 2.41 kPa, and 4.85 kPa. Each yield locus was obtained with four normal stresses. Linear backward extrapolation of the locus to the *y*-axis of the σ:τ plot was used to obtain an estimate of powder cohesion, C. Mohr circle analysis was used to determine the values of unconfined yield stress, σ_y, and major consolidation stress, σ_c; the σ_c:σ_y pairs for each material constitute its Powder Flow Function. The Brookfield shear cell apparatus has a capability for measuring bulk density *in situ*, ρ_B, and the values at each different preconsolidation stress are listed in Table 1.

Table 1
Particle size $d_{50,M}$, surface–volume mean diameter, $d_{32,M}$, and bulk density at different preconsolidation stresses for model lactose powders.

Model lactose	Particle size (μm)		Bulk density, ρ_B (kg m^{-3}) Preconsolidation stress, σ_{pre} (kPa)				
	$d_{50,M}$	$d_{32,M}$	0.31	0.61	1.20	2.41	4.85
LP4	34.5	4.4	591	644	708	782	844
LM7	37.4	5.6	605	658	723	784	841
LM8	56.1	9.4	656	705	758	813	862
LM1	108.5	9.7	786	821	869	911	951
LM9	64.4	10.3	681	724	773	821	863
LM4	139.5	17.8	790	828	865	903	936
LP2	139.0	19.8	832	862	895	928	954
LM2	112.9	20.0	762	789	820	851	875
LM3	143.2	27.8	788	804	823	843	862
LM5	251.8	31.9	834	853	874	896	912
LP1	257.0	37.1	902	924	949	973	995
LM6	242.0	42.1	807	823	844	863	881
LP3	263.4	60.6	871	879	891	907	922

3. Background and modeling

3.1. Coulomb yield criterion

According to the Coulomb criterion, Eq. (1), the shear stress required to fracture a consolidated bed of powder is the sum of the frictional contact stresses involved in the sliding between particles, term $\mu\sigma$, and the cohesion, C.

$$\tau = \mu\sigma + C \tag{1}$$

The processes taking place when a powder bed undergoes shear deformation are complex, and have been described and reviewed by Schulze [7]. Shear failure occurs in a zone, not a simple plane, and the thickness of the shear zone is apparently dependent on mean particle size, approximately 5–20 particle diameters for particles greater than ~100 μm and ~200 particle diameters for very fine powder. When a bed of compacted particles is sheared, the particles in the shear zone have to react against the applied normal stress to free themselves enough so they can force themselves past one another; this relative movement results in bed dilation, which affects the maximum shear stress at incipient flow [7].

Following Molerus [8] who derived a theoretical expression for cohesion in an unconsolidated powder, cohesion in a polydisperse bulk powder under compaction is expected to be related to the number of points of interparticle contact, and hence co-ordination number. The number of particle–particle contacts is not directly measureable, but is expected to depend on the particle surface area per unit volume. For a shear zone of constant cross-sectional area, the number of particle contacts will depend on the zone thickness which could be deduced if the following were known: (i) the bulk density as a function of preconsolidation stress, and (ii) the bed dilation. For (i), it is assumed that the bulk density measured in static tests is predictive of the bulk density when the bed is sheared at the same preconsolidation stress. For (ii), direct measurement of bed dilation is not available to us. However, as the cohesion C relates to the forces that must be overcome before flow commences, the dilation will produce a normal reaction stress which is assumed to be equal to the preconsolidation stress; thus C is postulated to be a function of particle surface area per unit volume of the powder bed and the dilation force per unit area across the shear zone, per Eq. (2).

$$C \propto f(\text{Surface area per unit volume, Dilation force per unit area}) \tag{2}$$