Available online at www.sciencedirect.com

ScienceDirect

Procedia Engineering 102 (2015) 218 – 225

Procedia Engineering

www.elsevier.com/locate/procedia

The 7th World Congress on Particle Technology (WCPT7)

Correlation between Powder Flow Properties Measured by Shear Testing and Hausner Ratio

Horng Yuan Saw[a,b], Clive E. Davies[a,b,]*, Anthony H.J. Paterson[a], Jim R. Jones[a]

[a] School of Engineering and Advanced Technology, Massey University, Palmerston North 4442, New Zealand
[b] Riddet Institute, Massey University, Palmerston North 4442, New Zealand

Abstract

Shear testing provides rigorous estimates of flow properties relevant to the characterization, handling, and processing of powders, and is a necessary test procedure in the formal design of powder storage facilities. However, despite the automation of modern test equipment, it can be time consuming and expensive. In contrast, measurement of bulk density is straightforward and less laborious, and tapping devices are cheaper. Here we explore the relationship between Hausner ratio and cohesion and also examine correlation between Hausner ratio, σ_c/σ_y, and σ_{pre} for a suite of 13 milled and 2 spray-dried lactose powders, 3 sand samples and 3 samples of refractory dust; Hausner ratio is the ratio of tapped bulk density to loose bulk density, σ_c is major consolidation stress, σ_y is unconfined yield stress and σ_{pre} is preconsolidation stress. Cohesion and flow function were measured with an annular shear cell at values of σ_{pre} up to 5 kPa. Loose poured bulk density was measured following a modified New Zealand standard and tapped density measurement was based on a method for dry dairy products and the European Pharmacopoeia; Hausner ratio at 1250 taps was used. Our results show that cohesion at σ_{pre} of 0.31 kPa, 0.61 kPa, 1.20 kPa, 2.41 kPa, and 4.85 kPa correlates linearly with Hausner ratio; the slope and intercept of the correlation are functions of σ_{pre}. A plot of σ_c/σ_y against Hausner ratio shows an exponential decay trend and regression yields two fitting parameters that correlate well with σ_{pre}. These correlations are potentially useful for assessing flow characteristics when shear testing cannot be performed.

Selection and peer-review under responsibility of Chinese Society of Particuology, Institute of Process Engineering, Chinese Academy of Sciences (CAS)

Keywords: Bulk density; cohesion; Hausner ratio; powder flow function; tapped density

* Corresponding author. Tel.: +64 63569099; fax: +64 63505604.
E-mail address: C.Davies@massey.ac.nz

Selection and peer-review under responsibility of Chinese Society of Particuology, Institute of Process Engineering, Chinese Academy of Sciences (CAS)
doi:10.1016/j.proeng.2015.01.132

Nomenclature

C	cohesion (Pa)
C^*	estimated cohesion (Pa)
d^*_{32}	surface-volume mean particle diameter (m) calculated with the Mastersizer data using bins equivalent to a BS 410 full sieve analysis; the powder in the range of 0–38 μm is grouped together and assigned a mean diameter of 19 μm
H_R	Hausner ratio (-)
$H_{R,1250}$	Hausner ratio at 1,250 taps (-)
k_{C1}, k_{C2}	fitting parameters of Eq. 1 [units according to usage]
k_{F1}, k_{F2}	fitting parameters of Eq. 5 [units according to usage]
Greek letters	
ρ_0	loose poured bulk density (kg m^{-3})
ρ_{tap}	tapped density (kg m^{-3})
σ_c	major consolidation stress (Pa)
σ_D	major stress developed in a dome or pipe (Pa)
σ_{pre}	preconsolidation stress (Pa)
σ_y	unconfined yield stress (Pa)
σ^*_c	estimated major consolidation stress (Pa)
σ^*_y	estimated unconfined yield stress (Pa)

1. Introduction

Knowledge of powder flowability is important to the handling and processing of powders across many different industries. The shear testing advocated by Jenike [1], which is necessary in the formal design of powder storage facilities, has been used to provide rigorous estimates of flow properties such as yield locus, cohesion, C, the ratio of major consolidation stress to unconfined yield stress, σ_c/σ_y, and Powder Flow Function. Shear cells can be expensive, and the shear testing protocol can be laborious and time consuming despite the automation of modern and computerized test devices.

A more straightforward and convenient way to assessing powder flowability is the measurement and use of Hausner ratio, H_R, see for example [2]; H_R is the ratio of tapped density, ρ_{tap}, to loose poured bulk density, ρ_0, which can be measured with various standards such as the European Pharmacopoeia [3] and also non-standard methods. In comparison with shear cells, tapping devices are cheaper and easier to operate. But from a scientific point of view, a major drawback is the empiricism of H_R; it is only a single index that provides limited information on powder flowability.

In seeking the connections between bulk densities and powder flow properties measured by shear testing, Stanley-Wood et al. [4] investigated the relationships between H_R and σ_c/σ_y at 3 kPa; the stress value of 3 kPa was based on Jenike [1]. With their data sets, a "*complicated logarithmic relationship*" between H_R and σ_c/σ_y was observed. The value of H_R was also constant at 1.25 with powders that are "*free flowing*". The nature of their work was preliminary, and no correlation was proposed.

In this paper, we explore the relationships between H_R, C, σ_c/σ_y, and preconsolidation stress, σ_{pre}, for samples of milled and spray-dried lactose powders, sand, and refractory dust. Emphasis is on the correlation between C and H_R, and σ_c/σ_y and H_R. Our motivation is that with such correlations, independent measurement of H_R can provide quick assessments of C and σ_c/σ_y, and hence flowability when shear testing facilities are not accessible.

2. Materials and methods

2.1. Materials

A total of 13 milled lactose powders, 2 spray-dried lactose powders, 3 sand samples, and 3 refractory dust samples were used; each powder was given a code as listed in Table 1. Information on the preparation of the milled lactose powders by sieving LM1 or LP1 has been reported earlier [5, 6]. The spray-dried lactose samples were prepared with a similar procedure from a commercial product (SuperTab®, DMV-Fonterra Excipients, New Zealand). Sand S1 and refractory dust RD1 were used as received; the other sand and refractory dust samples were prepared from S1 and RD1 respectively with the procedure outlined in [5, 6]. Listed in Table 1 are the values of d^*_{32}, which is the surface-volume mean particle diameter (m) measured by the laser diffraction method (Mastersizer 2000, Malvern Instruments Ltd., UK) and calculated with the Mastersizer data using bins equivalent to a BS 410 full sieve analysis; the powder in the range of 0–38 μm is grouped together and assigned a mean diameter of 19 μm, see [5, 6].

2.2. Shear testing

Cohesion, σ_c/σ_y, and Powder Flow Function were measured at σ_{pre} of 0.31 kPa, 0.61 kPa, 1.20 kPa, 2.41 kPa and 4.85 kPa with an annular shear cell (Brookfield Engineering Laboratories Inc., USA) under ambient conditions (20–24°C, 36–54% relative humidity); the detailed experimental protocol is available elsewhere, see [5, 6]. With information on Powder Flow Function and Jenike's arbitrary powder flow divisions, namely *very cohesive* when $\sigma_c/\sigma_D<2$, *cohesive* when $2<\sigma_c/\sigma_D<4$, *easy flowing* when $4<\sigma_c/\sigma_D<10$, and *free flowing* when $10<\sigma_c/\sigma_D$ [1], the flowability of each powder was inferred; σ_D is the major stress developed in a dome or pipe (Pa). Consistent with previous work [5, 6], the σ_c value of ~2 kPa, which corresponded to σ_{pre} of 1.2 kPa, was considered.

2.3. Measurements of loose poured bulk density and tapped density

Loose poured bulk density was measured with a modified New Zealand standard method [7]; further details are given in [8]. Tapped density was measured with a method for dry dairy products [9] and the number of taps was 1,250 following the European Pharmacopoeia [3]; further details are available in [8].

3. Results and discussion

The d^*_{32} of the powders used and their respective flowability at σ_{pre}=1.20 kPa are given in Table 1; the value of σ_{pre}=1.20 kPa is chosen based on precedent work with milled lactose powders [5, 6]. At this σ_{pre} and with our data sets, the σ_c/σ_y of the selected powders falls into either one of the four Jenike's arbitrary flow divisions. When σ_{pre} is above 1.20 kPa, the powders are consistently *easy flowing* or *free flowing*.

Fig. 1 shows C at σ_{pre}=1.20 kPa plotted against $H_{R,1250}$, the Hausner ratio at 1,250 taps; the plot seems linear and similar trends are observed with the data sets at σ_{pre} of 0.31 kPa, 0.61 kPa, 2.41 kPa, and 4.85 kPa (results not shown). By linear regression, Eq. 1 is obtained; k_{C1} and k_{C2} are fitting parameters. In Fig. 2, k_{C1} is plotted against $\log(\sigma_{pre})$ and in Fig. 3 k_{C2} against $\log(\sigma_{pre})$; both figures show apparent linear trends and Eq. 2 and Eq. 3 are obtained. The substitution of Eq. 2 and Eq. 3 into Eq. 1 yields Eq. 4; C^* is the estimated cohesion.

$$C = k_{C1} H_{R,1250} + k_{C2} \tag{1}$$

$$k_{C1} = 0.6096 \log \sigma_{pre} + 0.4695 \tag{2}$$

$$k_{C2} = -0.7250 \log \sigma_{pre} - 0.5180 \tag{3}$$

$$C^* = \log\left(\sigma_{pre}{}^{0.6096 H_{R,1250} - 0.7250}\right) + 0.4695 H_{R,1250} - 0.5180 \tag{4}$$

Table 1. Surface-volume mean particle diameter and flowability of powders at σ_{pre} of 1.2 kPa according to Jenike's arbitrary powder flow divisions [1].

Powders	d^*_{32} (μm)	Powder flowability at σ_{pre} of 1.20 kPa
Unsieved milled lactose		
LP4	28.9	*Very cohesive*
LM1	58.0	*Cohesive*
LP1	150.8	*Free flowing*
Sieved milled lactose		
LM7	29.9	*Cohesive*
LM8	39.3	*Cohesive*
LM9	43.3	*Cohesive*
LM4	65.1	*Easy flowing*
LM2	73.4	*Easy flowing*
LP2	83.6	*Easy flowing*
LM3	110.7	*Free flowing*
LM5	113.4	*Free flowing*
LM6	163.7	*Free flowing*
LP3	223.0	*Free flowing*
Spray-dried lactose		
LT1	35.8	*Easy flowing*
LT2	102.2	*Free flowing*
Sand		
S3	28.7	*Easy flowing*
S1	40.0	*Easy flowing*
S2	76.9	*Free flowing*
Refractory dust		
RD3	23.3	*Easy flowing*
RD1	41.5	*Easy flowing*
RD2	66.6	*Free flowing*

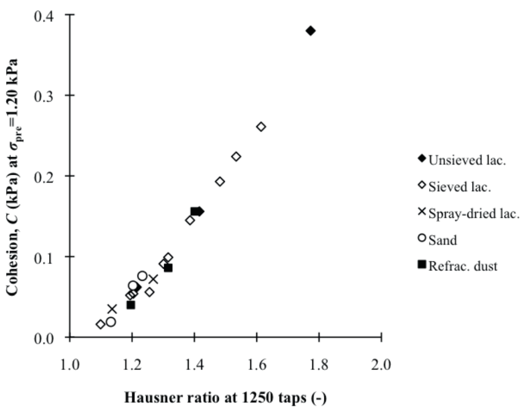

Fig. 1. Plot of C at σ_{pre}=1.20 kPa versus $H_{R,1250}$ for milled and spray-dried lactose powders, sand, and refractory dust.

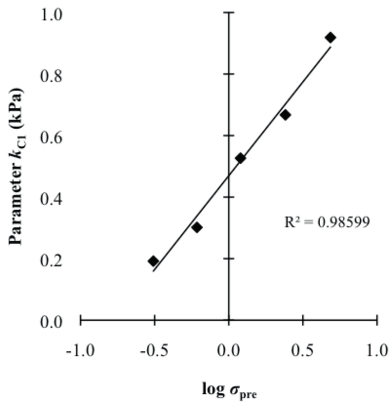

Fig. 2. Plot of k_{C1} versus log(σ_{pre}).

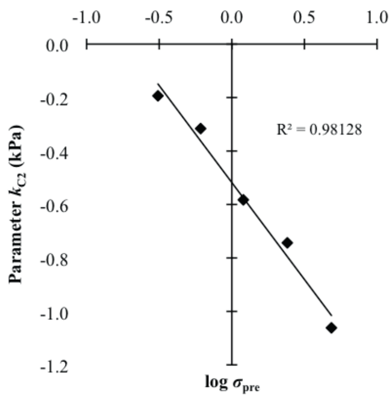

Fig. 3. Plot of k_{C2} versus log(σ_{pre}).

Fig. 4 shows a plot of σ_c/σ_y at σ_{pre}=1.20 kPa against $H_{R,1250}$; similar trends are found at σ_{pre} of 0.31 kPa, 0.61 kPa, 2.41 kPa, and 4.85 kPa and these results are not presented. Regression of the data in Fig. 4 gives Eq. 5; k_{F1} and k_{F2} are fitting parameters. Parameter k_{F1} is plotted against σ_{pre}^2 in Fig. 4, and k_{F2} against σ_{pre} in Fig. 5; both figures demonstrate apparent linear trends, giving Eq. 6 and Eq. 7 respectively. Eq. 8 is obtained when Eq. 6 and Eq. 7 are substituted into Eq. 5; σ^*_c/σ^*_y is the estimated ratio of major consolidation stress to unconfined yield stress.

$$\frac{\sigma_c}{\sigma_y} = k_{F1} H_{R,1250}^{\;-k_{F2}} \tag{5}$$

$$k_{F1} = 13.8531\sigma_{pre}^{\;2} + 9.0954 \tag{6}$$

$$k_{F2} = 0.9678\sigma_{pre} + 4.3098 \tag{7}$$

$$\frac{\sigma^*_c}{\sigma^*_y} = \frac{13.8531\sigma_{pre}^2 + 9.0954}{H_{R,1250}^{\;0.9678\sigma_{pre}+4.3098}} \tag{8}$$

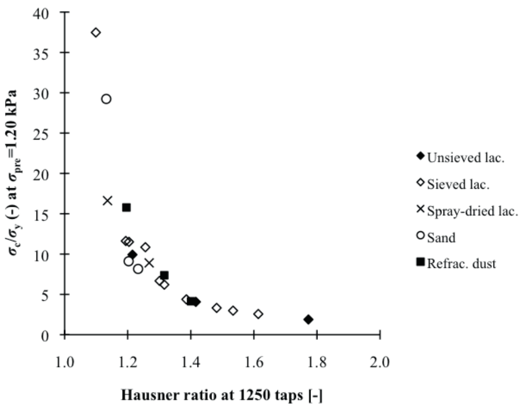

Fig. 4. Plot of σ_c/σ_y at σ_{pre}=1.20 kPa versus $H_{R,1250}$ for milled and spray-dried lactose powders, sand, and refractory dust.

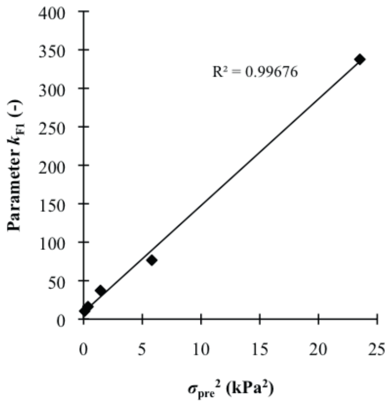

Fig. 5. Plot of k_{F1} versus σ_{pre}^2.

Listed in Table 2 are the range of correlation error for Eq. 4, $(C^*-C)/C$, and for Eq. 8, $[(\sigma^*_c/\sigma^*_y)-(\sigma_c/\sigma_y)]/(\sigma_c/\sigma_y)$. With reference to powders that are *very cohesive* and *cohesive* at σ_{pre}=1.20 kPa, the correlation error is relatively small and hence considered acceptable. However for *easy flowing* and *free flowing* powders, the correlation error is high; we believe this is mainly attributed to the scatter in the data sets. We have begun to address this using the milled lactose data sets in our latest communication [6].

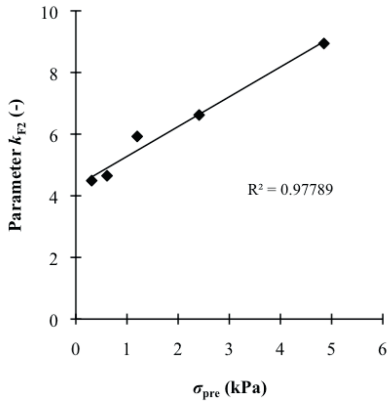

Fig. 6. Plot of k_{F2} versus σ_{pre}.

Table 2. Range of correlation error, $(C^*-C)/C$ for Eq. 4 and $[(\sigma^*_c/\sigma^*_y)-(\sigma_c/\sigma_y)]/(\sigma_c/\sigma_y)$ for Eq. 8.

Powder flowability at σ_{pre} of 1.20 kPa based on Jenike's flow divisions	Range of correlation error, $(C^*-C)/C$ (%)	Range of correlation error, $[(\sigma^*_c/\sigma^*_y)-(\sigma_c/\sigma_y)]/(\sigma_c/\sigma_y)$ (%)
Very cohesive	–18.3% to –1.7	–39.5% to –9.3
Cohesive	–11.0% to +28.7	–45.9% to +26.7
Easy flowing	–34.0% to +90.4	–13.6% to +127.2
Free flowing	–234.9% to +244.5	–76.7% to +134.9

4. Conclusions

The correlation between C and σ_c/σ_y measured by shear testing at σ_{pre} below 5 kPa and $H_{R,1250}$ was investigated; this work was inspired by and seeks to extend the work by Stanley-Wood et al. [4]. Eq. 4 and Eq. 8 are derived and proposed to give estimates of C and σ_c/σ_y for milled and spray-dried lactose powders, sand, and refractory dust. The correlation error is small with powders that are categorized as *very cohesive* and *cohesive* according to Jenike's criteria for powder flowability, but high with *easy flowing* and *free flowing* powders; hence caution has to be taken in the use of Eq. 4 and Eq. 8.

Acknowledgement

A Ph.D. scholarship from the Riddet Institute is acknowledged.

References

[1] A.W. Jenike, Storage and flow of solids, Bulletin No. 123 of Utah Engineering Experiment Station 53 (1964).
[2] E.C. Abdullah, D. Geldart, The use of bulk density measurements as flowability indicators, Powder Technol. 102 (1999) 151–165.
[3] A. Schüssele, A. Bauer-Brandl, Note on the measurement of flowability according to the European Pharmacopoeia, Int. J. Pharm. 257 (2003) 301–304.
[4] N. Stanley-Wood, M. Sarrafi, Z. Mavere, M. Schaefer, The relationships between powder flowability, particle re-arrangement, bulk density and Jenike failure function, Adv. Powder. Technol. 4 (1993) 33–40.
[5] H.Y. Saw, C.E. Davies, J.R. Jones, G. Brisson, A.H.J. Paterson, Cohesion of lactose powders under low consolidation stresses, Adv. Powder Technol. 24 (2013) 796–800.
[6] H.Y. Saw, C.E. Davies, J.R. Jones, A.H.J. Paterson, Shear testing of lactose powders: The influence of consolidation stress and particle size on bulk density and estimated cohesion, Adv. Powder Technol. (2014) dx.doi.org/10.1016/j.apt.2014.05.009.

[7] SANZ, Method for determining voids content, flow time and percentage oversize material in sand, in: NZS 3111:1986 – Methods of test for water and aggregate for concrete, Standards Association of New Zealand, Wellington, 1986, pp. 44–45.
[8] H.Y. Saw, C.E. Davies, A.H.J. Paterson, J.R. Jones, The influence of particle size distribution and tapping on the bulk density of milled lactose powders, Proceedings of CHEMECA 2013 Conference, Brisbane, Australia, 2013.
[9] Niro, Method No. A2a – Packed bulk density by the Niro method for milk powders and protein products, in: Analytical methods for dry milk products, A/S Niro Atomizer, Copenhagen, 1978, pp. 2.3.1–2.3.4.