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

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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 and cohesion 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, $\sigma_c$ is major consolidation stress, $\sigma_y$ is unconfined yield stress and $\sigma_{pre}$ is preconsolidation stress. Cohesion and flow function were measured with an annular shear cell at values of $\sigma_{pre}$ 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 $\sigma_{pre}$ 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 $\sigma_{pre}$. A plot of $\sigma_c/\sigma_y$ against Hausner ratio shows an exponential decay trend and regression yields two fitting parameters that correlate well with $\sigma_{pre}$. These correlations are potentially useful for assessing flow characteristics when shear testing cannot be performed.

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

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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, $\sigma_c/\sigma_y$, 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, $\rho_{tap}$, to loose poured bulk density, $\rho_0$, 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 $\sigma_c/\sigma_y$ 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 $\sigma_c/\sigma_y$ 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 $H_R$, $C$, $\sigma_c/\sigma_y$, and preconsolidation stress, $\sigma_{pre}$, for samples of milled and spray-dried lactose powders, sand, and refractory dust. Emphasis is on the correlation between $C$ and $H_R$, and $\sigma_c/\sigma_y$ and $H_R$. Our motivation is that with such correlations, independent measurement of $H_R$ can provide quick assessments of $C$ and $\sigma_c/\sigma_y$, 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, $\sigma_c/\sigma_y$ and Powder Flow Function were measured at $\sigma_{pre}$ 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 $\sigma_c/\sigma_y<2$, cohesive when $2<\sigma_c/\sigma_y<4$, easy flowing when $4<\sigma_c/\sigma_y<10$, and free flowing when $10<\sigma_c/\sigma_y$ [1], the flowability of each powder was inferred; $\sigma_0$ is the major stress developed in a dome or pipe (Pa). Consistent with previous work [5, 6], the $\sigma_c$ value of ~2 kPa, which corresponded to $\sigma_{pre}$ 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 $\sigma_{pre}=1.20$ kPa are given in Table 1; the value of $\sigma_{pre}=1.20$ kPa is chosen based on precedent work with milled lactose powders [5, 6]. At this $\sigma_{pre}$ and with our data sets, the $\sigma_c/\sigma_y$ of the selected powders falls into either one of the four Jenike’s arbitrary flow divisions. When $\sigma_{pre}$ is above 1.20 kPa, the powders are consistently easy flowing or free flowing.

Fig. 1 shows $C$ at $\sigma_{pre}=1.20$ 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 $\sigma_{pre}$ 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.

\[
C = k_{C1}H_{R,1250} + k_{C2}
\]  \hspace{1cm} (1)

\[
k_{C1} = 0.6096\log(\sigma_{pre}) + 0.4695
\]  \hspace{1cm} (2)

\[
k_{C2} = -0.7250\log(\sigma_{pre}) - 0.5180
\]  \hspace{1cm} (3)

\[
C^* = \log\left(\sigma_{pre}^{0.6096H_{R,1250}^{0.7250}}\right) + 0.4695H_{R,1250} - 0.5180
\]  \hspace{1cm} (4)
Table 1. Surface-volume mean particle diameter and flowability of powders at $\sigma_{pre}$ of 1.2 kPa according to Jenike’s arbitrary powder flow divisions [1].

| Powders                  | $d^{*32}$ (μm) | Powder flowability at $\sigma_{pre}$ 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 $\sigma_{pre}$=1.20 kPa versus $H_{R,1250}$ for milled and spray-dried lactose powders, sand, and refractory dust.
Fig. 4 shows a plot of $\sigma_c/\sigma_y$ at $\sigma_{pre}=1.20$ kPa against $H_{R,1250}$; similar trends are found at $\sigma_{pre}$ 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 $\sigma_{pre}^2$ in Fig. 4, and $k_{F2}$ against $\sigma_{pre}$ 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; $\sigma^*/\sigma^*_y$ is the estimated ratio of major consolidation stress to unconfined yield stress.

$$\frac{\sigma_c}{\sigma_y} = k_{F1} H_{R,1250}^{-k_{F2}}$$  \hspace{1cm} (5)

$$k_{F1} = 13.8531 \sigma_{pre}^2 + 9.0954$$  \hspace{1cm} (6)

$$k_{F2} = 0.9678 \sigma_{pre} + 4.3098$$  \hspace{1cm} (7)
\[
\frac{\sigma_{c}^{*}}{\sigma_{y}^{*}} = \frac{13.8531 \sigma_{pre}^{2} + 9.0954}{H_{R,1250}^{0.9678 \sigma_{pre} + 4.3098}}
\]  

(8)

Fig. 4. Plot of \(\sigma_{c}/\sigma_{y}\) at \(\sigma_{pre} = 1.20\) kPa versus \(H_{R,1250}\) for milled and spray-dried lactose powders, sand, and refractory dust.

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 \(\sigma_{pre} = 1.20\) 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 $k_F^2$ versus $\sigma_{\text{pre}}$.

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

| Powder flowability at $\sigma_{\text{pre}}$ of 1.20 kPa based on Jenike’s flow divisions | Range of correlation error, $(C^* - C)/C$ (%) | Range of correlation error, $((\sigma^c/\sigma^s) - (\sigma_c/\sigma_y))/((\sigma_c/\sigma_y))$ (%) |
|-----------------------------------------------|-----------------------------------------------|------------------------------------------------|
| 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 $\sigma_c/\sigma_y$ measured by shear testing at $\sigma_{\text{pre}}$ 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 $\sigma_c/\sigma_y$ 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.

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