

Received:
15 October 2018
Revised:
7 January 2019
Accepted:
23 January 2019

Cite as: David J. Sun,
Changquan Calvin Sun.
Proportionality between
powder cohesion and
unconfined yield strength
from shear cell testing.
Heliyon 5 (2019) e01171.
doi: [10.1016/j.heliyon.2019.e01171](https://doi.org/10.1016/j.heliyon.2019.e01171)



Proportionality between powder cohesion and unconfined yield strength from shear cell testing

David J. Sun^a, Changquan Calvin Sun^{b,*}

^a Nova Classical Academy, St. Paul, MN 55102, USA

^b Department of Pharmaceutics, College of Pharmacy, University of Minnesota, 308 Harvard St. S.E. Minneapolis, MN 55455, USA

* Corresponding author.

E-mail address: sunx0053@umn.edu (C.C. Sun).

Abstract

From an analysis of the geometry of the yield locus and the Mohr's circle for determining unconfined yield strength (f_c) in shear cell testing, it has been shown that powder cohesion is proportional to f_c , where the proportionality constant is a function of angle of linearized yield locus, $(1-\sin\theta)/(2\cos\theta)$. While both parameters are routinely included in shear cell data, only one parameter is needed to characterize flow properties of a new powder.

Keywords: Chemical engineering, Pharmaceutical science

1. Introduction

Powder flowability is one of the critical material attributes that affect powder handling in several industries, including pharmaceutical manufacturing [1, 2]. For example, high speed tableting requires the powder to have “acceptable flow properties” for consistent die filling, tablet weight, and content uniformity [3]. Powder flow properties can be measured using different techniques [4, 5, 6], but shear cells are the most fundamental, versatile, and reliable technique [7]. Consequently, it has been

widely applied to characterize pharmaceutical powders to guide formulation development and optimization [8, 9, 10, 11].

A shear cell measurement can produce a number of useful parameters to describe powder flow properties [7, 12, 13, 14]. The unconfined yield strength (f_c) is the most commonly used parameter to quantify powder flowability. A flowability index, ffc , defined as the ratio of major principal stress (σ_1) to f_c [15, 16], has been used to quantify powder flowability. In general, a powder with a higher f_c at the same σ_1 tends to exhibit poorer powder flow. Recently, it was observed that f_c and powder cohesion, C , exhibit a peculiarly similar relationship with σ_1 (Fig. 1) [15], indicating a possible intrinsic proportional relationship between these two parameters. A proportionality between the two parameters is also suggested by the observation of nearly perfect linear relationship between experimental f_c and C for 25 fine powders [17]. Here, we derive a mathematical relationship between the two parameters to explain this phenomenon.

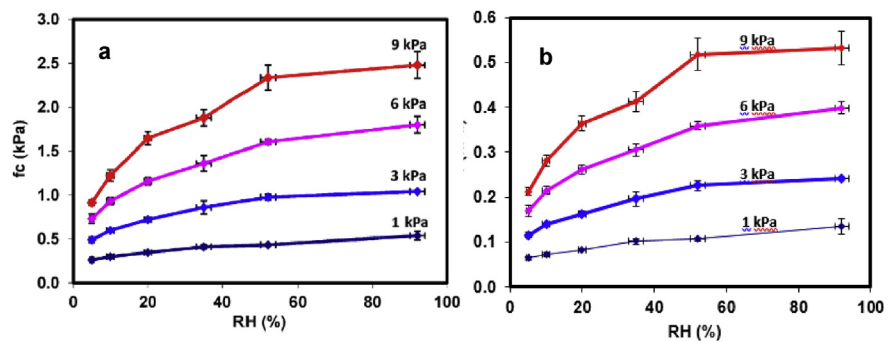


Fig. 1. a) Unconfined yield strength and b) cohesion plots of microcrystalline cellulose equilibrated at different relative humidities. Data were collected at four pre-shear consolidation stresses (Ref. [15]).

2. Theory

In the classical approach of analyzing powder flowability using a shear cell, a yield locus is experimentally determined by measuring the maximum shear stress the powder can sustain under a given normal stress [18]. Repeating the measurement at different normal stresses delineates the yield locus. According to the shear cell theory, C is the intercept of a yield locus with the shear stress (τ) axis. A series of semi-circles that are below and tangent to the yield locus can be drawn to represent the stress distribution inside the powder under different external stress conditions. The semicircle passing the origin crosses the normal stress (σ_n) axis at f_c (Fig. 2) [14]. An inspection of these basic elements of the figure indicates these two parameters can be related by taking a trigonometric approach.

To solve the problem, we can connect the tangent point, D, and the center of the circle, B. The radius BD forms a right angle with the yield locus line. If the yield locus

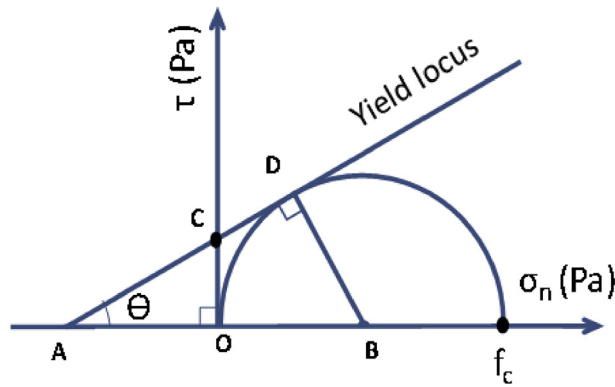


Fig. 2. Illustration of the relationship between cohesion, C , and f_c , derived from a yield locus.

is extrapolated to intersect with the σ_n axis at point A, angle θ is identified. In cases where the yield locus is not strictly linear, this angle corresponds to the angle of linearized yield locus [14]. The two right triangles, ADB and AOC, are similar because they share the same angle, θ . Therefore, Eq. (1) is written by applying the rule of proportionality between similar triangles.

$$\frac{AB}{AC} = \frac{BD}{CO} \quad (1)$$

Expressing the lengths of each side in terms of f_c and C yields Eq. (2).

$$\left(\frac{\frac{C}{\tan\theta} + \frac{f_c}{2}}{\frac{C}{\sin\theta}} \right) = \left(\frac{\frac{f_c}{2}}{C} \right) \quad (2)$$

We can substitute $\tan(\theta)$ in Eq. (2) with $\sin(\theta)/\cos(\theta)$ and rearrange terms to get Eq. (3)

$$\left(C \frac{\cos\theta}{\sin\theta} + \frac{f_c}{2} \right) \cdot \left(\frac{\sin\theta}{C} \right) = \frac{f_c}{2C} \quad (3)$$

Solving for C by rearranging terms in Eq. (3) leads to Eq. (4),

$$C = f_c \frac{(1 - \sin\theta)}{2\cos\theta} \quad (4)$$

Eq. (4) explicitly indicates a proportionality between f_c and C , with the proportionality constant, $(1 - \sin\theta)/(2\cos\theta)$. This equation was recently suggested but derivation was not given [19]. The proportionality between C and f_c explains the similar shape of f_c and C plots shown in Fig. 1. This relation is consistent with the observed proportionality between f_c and C for the same material under different initial consolidation stresses. The proportionality constants differed for different materials, which may be attributed to their different θ values [20].

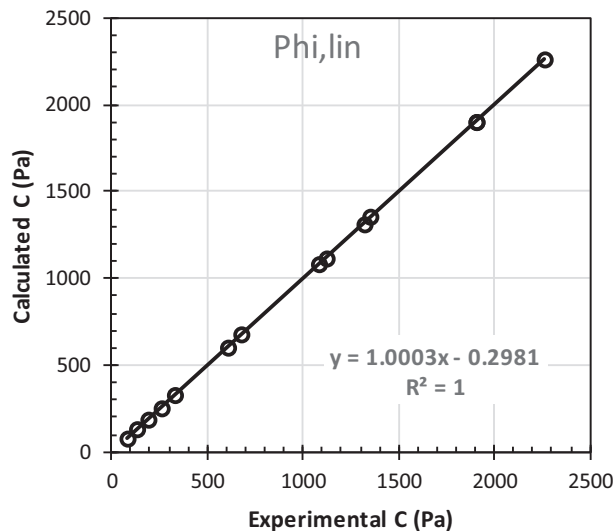


Fig. 3. Relationship between C values obtained from extrapolating experimental yield locus to zero normal stress and those calculated from Eq. (4).

3. Results & discussion

Proportionality in Eq. (4) is a function of θ , which is different among powders. To validate this relationship, we used shear cell data of powders exhibiting a wide range of flow properties tested under different normal stresses, i.e., limestone reference powder for shear cell (pre-shear normal stresses of 3, 6, 9, 15 kPa), microcrystalline cellulose (Avicel Ph105 and Avicel PH102, pre-shear normal stresses of 1, 3, 6, 9 kPa). Data were collected using a rotational shear cell (RST-XS, Dietmar Schulze, Wolfenbüttel, Germany). The angle of linearized yield locus was used to calculate the proportionality constant. Subsequently, for each experimentally determined f_c value, a corresponding C value was predicted from Eq. (4), which was then plotted against C values obtained by extrapolating the yield locus (Fig. 3). The linear regression equation has a slope of essentially unity (1.0003) and an intercept of essentially zero (0.2981 Pa). Thus, the regression line closely resembles the ideal line, which suggests the validity of the derived relationship between f_c and C . Eq. (4) is valid as long as the yield locus between the y-intercept and the tangency point, i.e., CD in Fig. 2, is linear. However, when the yield locus is curved, deviations from Eq. (4) are expected. Therefore, a gross deviation from the proportionality is a clear indication of curvature of the yield locus in the pressure range below the tangency point, D, in Fig. 2. This occurs usually when the powder is very cohesive and, hence, flows poorly.

4. Conclusion

The similarity between cohesion and flow function plots derived from shear cell data is explained by the proportionality between f_c and C values derived from a given

yield locus, where the proportionality constant is equal to $(1-\sin\theta)/(2\cos\theta)$. Given the proportionality, only one of the two flow parameters needs to be analyzed to compare flowability among different powders or to predict flow behavior of a given powder.

Declarations

Author contribution statement

David J. Sun: Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Changquan Calvin Sun: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

Funding statement

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

References

- [1] J.K. Prescott, R.A. Barnum, On powder flowability, *Pharmaceut. Technol.* 24 (10) (2000) 60–84. <https://www.powderbulk.com/enews/2014/whitepaper/jenike042014.pdf>.
- [2] E.C. Abdullah, A.M. Salam, A.R. Aziz, Cohesiveness and flowability properties of silica gel powder, *Phys. Int.* 1 (2010) 16–21.
- [3] C.C. Sun, Setting the bar for powder flow properties in successful high speed tableting, *Powder Technol.* 201 (2010) 106–108.
- [4] USP Convention, <1174> Powder Flow [Harmonization] USP 29/NF 24, https://www.usp.org/sites/default/files/usp/document/harmonization/generalchapter/g05_pf_30_6_2004.pdf.

- [5] S.A. Mohammed, E.C. Abdullah, D. Geldart, A.A.A. Raman, Measuring powder flowability with a modified Warren Spring cohesion tester, *Particuology* 9 (2011) 148–154.
- [6] V. Garg, S.S. Mallick, P. Garcia-Trinanes, R.J. Berry, An investigation into the flowability of fine powders used in pharmaceutical industries, *Powder Technol.* 336 (2018) 375–382.
- [7] USP Convention, <1063> Shear cell methodology for powder flow testing. USP 39-NF 34, <https://www.uspnf.com/>.
- [8] K. Kunnath, Z. Huang, L. Chen, K. Zheng, R. Davé, Improved properties of fine active pharmaceutical ingredient powder blends and tablets at high drug loading via dry particle coating, *Int. J. Pharm.* 543 (2018) 288–299.
- [9] S. Paul, S.-Y. Chang, J. Dun, W.-J. Sun, K. Wang, P. Tajarobi, C. Boissier, C.C. Sun, Comparative analyses of flow and compaction properties of diverse mannitol and lactose grades, *Int. J. Pharm.* 546 (2018) 39–49.
- [10] T. Swize, F. Osei-Yeboah, M.L. Peterson, P. Boulas, Impact of shear history on powder flow characterization using a ring shear tester, *J. Pharm. Sci.* (2018).
- [11] S.V. Søgaaard, T. Pedersen, M. Allesø, J. Garnaes, J. Rantanen, Evaluation of ring shear testing as a characterization method for powder flow in small-scale powder processing equipment, *Int. J. Pharm.* 475 (1) (2014) 315–323.
- [12] D. Schulze, in: Berlin (Ed.), *Powder and Bulk Solids, Behaviour, Characterization, Storage and Flow*, Springer, New York, 2007.
- [13] P.D. Jager, T. Bramante, P.E. Luner, Assessment of pharmaceutical powder flowability using shear cell-based methods and application of Jenike's methodology, *J. Pharm. Sci.* 104 (11) (2015) 3804–3813.
- [14] L. Shi, S. Chatteraj, C.C. Sun, Reproducibility of flow properties of micro-crystalline cellulose — Avicel PH102, *Powder Technol.* 212 (2011) 253–257.
- [15] C.C. Sun, Quantifying effects of moisture content on flow properties of micro-crystalline cellulose using a ring shear tester, *Powder Technol.* 289 (2016) 104–108.
- [16] D. Schulze, A. Wittmaier, Flow properties of highly dispersed powders at very small consolidation stresses, *Chem. Eng. Technol.* 26 (2003) 133–137.
- [17] S.S. Mallick, L. Rohilla, V. Garg, G. Setia, Modeling flow properties of fine dry powders using particle morphological properties and its effects on geometry of fly ash evacuation hoppers, *Part. Sci. Technol.* 36 (2018) 464–472.

- [18] E.N. Hiestand, S.C. Valvani, C.B. Peot, E.P. Strzelinski, J.F. Glasscock, Shear cell measurements of powders: determination of yield loci, *J. Pharm. Sci.* 62 (1973) 1513–1517.
- [19] L.Y. Leung, C. Mao, I. Srivastava, P. Du, C.-Y. Yang, Flow function of pharmaceutical powders is predominantly governed by cohesion, not by Friction coefficients, *J. Pharm. Sci.* 106 (7) (2017) 1865–1873.
- [20] Y. Wang, S. Koynov, B.J. Glasser, F.J. Muzzio, A method to analyze shear cell data of powders measured under different initial consolidation stresses, *Powder Technol.* 294 (2016) 105–112.