Measuring powder flowability with a modified Warren Spring cohesion tester

Salam A. Mohammed, Ezzat C. Abdullah, Derek Geldart, Abdul Aziz A. Raman

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1. Introduction

Many chemical processes involve dealing with dry powders, as raw materials, intermediates or final products, making prediction of flow characteristics of great concern in bulk solid handling (Freeman, 2007). There are diverse ways in which powder flowability could be described, through the utilization of the different available measurement methods (Bell, 2001; Fitzpatrick & Ahrne, 2005). These are basically aerated and non-aerated techniques. Nonetheless, if there is a state of breakage, flow-blockage, or other production malfunction, powders could instantly change into a non-aerated state. Furthermore, for the period of storage in a hopper or storage tower, all powders are in a non-aerated state. Thus, in order to avoid flow-out halting from the hoppers, calculation of the non-aerated powder cohesivity becomes essential. The well known methods in the aerated category are the Hausner ratio (HR) (Abdullah & Geldart, 1999; Carr, 1965; Hausner, 1981; Riley & Hausner, 1970), angle of repose (AoR) (Cain, 2002; Wouters & Geldart, 1996) and behavior during fluidization (Bruni, Lettieri, Newton, & Barletta, 2007; Räsänen, Antikainen, & Yliruusi, 2003).

Significant contributions to measurements of non-aerated flow properties were made by Jenike (1964), who employed and modified techniques used in soil mechanics to shear non-aerated powders to investigate the powder strength. Jenike (1964) found that a powder may gain strength and develop flow problems when it is exposed to compressive stresses over time. Numerous researchers have used the Jenike shear tester to measure the flow properties of cement, powdered food and other granular materials (Fitzpatrick, Barringer, & Iqbal, 2004; Iqbal & Fitzpatrick, 2006; Kamath, Puri, & Manbeck, 1994; Schrämli, 1967; Teunou & Fitzpatrick, 2000). The Jenike shear cell technique is often preferred to other shear test methods, such as the triaxial method, because of its several advantages in hopper design (Kamath, Puri, & Manbeck, 1994). Many other shear testers have been developed over the years such as Schulze ring shear tester (SRST); Johanson Indimizer system (JIS) and the rotational split level shear tester (Peschl) (McClincy, 2005).

Orband and Geldart (1997) introduced the Warren Spring-Bradford cohesion tester (WSBCT) which is capable of measuring directly the cohesive strength of a powder. Verlinden (2000) developed an improved version of WSBCT as part of the work on...
characterization. The Jenike shear cell can also measure the cohesion strength but only indirectly through extrapolation of results from the shear cell.

In this work, flowability has been characterized using a cohesion tester called Warren Spring-University of Malaya cohesion tester (WSUMCT) and Hosokawa PT-S for porous (silica gel) and non-porous (ballotini) mixtures of industrial powders. All the powder mixtures were tested using the above methods to make a fair evaluation of their flowability. Powder mixtures were prepared by mixing fine and coarse components in known percentages in order to observe the effect of particle size on the flowability of silica gel and ballotini powders.

2. Materials and methods

2.1. Material

A porous silica gel powder (supplied by Merck, Germany) and non-porous ballotini 2000CP (supplied by Potters Industries Inc. Australia) were used in the present work. A series of powder samples having different mean particle sizes was prepared by mixing certain quantities of fine silica gel (9.4 µm) particles with coarse silica gel (60 µm) particles. The mass percentages of fines were 0, 1, 2, 5, 7, 10, 15, 20, 25, 30, 40 and 100%. The 28 µm fine ballotini powder was added to the 72 µm coarse ballotini particles to form 0, 10, 20, 30, 40, 60, 80 and 100% mass percentages of fines of non-porous powder mixtures. These samples were prepared by mixing the powders with a Kenwood mixer for 5 min and subsequently dried at 90°C for 3 h before each test was conducted.

Tables 1 and 2 display the flow and physical properties of the fine and coarse silica gel and ballotini powders. The mean particle size for each sample was measured using Malvern Mastersizer.

2.2. Equipment

2.2.1. Warren Spring-University of Malaya Cohesion Tester (WSUMCT)

The WSUMCT is a device that has been developed from the WSBCT. The main part of the WSBCT consists of a stainless-steel paddle wheel having eight thin vanes (50 mm inner diameter, 100 mm outer diameter and 12 mm height). After filling a Perspex cylindrical cell with 500 mL of powder and compacting the powder, the paddle is inserted into the sample. Torque is applied to the paddle via a calibrated spring, and the torque that is required to rotate the segmented disc of powder held within the vanes of the paddle is measured.

The powder tends to resist the torque applied by a geared electric motor via the vanes, and the torque is gradually increased to a value which is just sufficient to cause powder failure. That value is assumed to be the cohesion measured in the absence of any normal load applied to the powder (Geldart, Abdullah, & Verlinden, 2009).

Since it is difficult to observe the exact value of the torque at failure in the WSBCT, an effort was made to detect the powder failure through the use of electronics employing a torque sensor to measure the applied force which is directly linked to a computer via an interface module to give more accurate digital values (see Fig. 1). Modified Lab View module software was employed to record the sensor data during the shear test on the compacted powder by the vanes. Using WSUMCT, neither calibrated springs nor their calibration curves are required. The torque is now applied by a motor connected to a computer that displays digitally the torque values applied, whereas in the original WSBCT, the torque values applied are obtained by reading the angle at which failure occurs.

2.2.2. Hosokawa PT-S

The Hosokawa PT-S model, which is a compact machine, was used to measure AoR and HR as described in Sections 2.3.1 and 2.3.2.

Both particle size and particle size distribution play significant roles in flowability-related parameters such as bulk density, AoR and compressibility of bulk solids. Even a small change in particle size can cause significant alterations in the resulting flowability. Reduction in particle size often tends to decrease the flowability of a given granular material due to increased surface area per unit mass, therefore increasing the cohesive forces (Cain, 2002; Fitzpatrick & Ahrne, 2005).

Considerable research has been carried out to study the effect of particle size and shape on the flowability of powders. Geldart, Abdullah, Hassanpour, Nwoke, and Wouters (2006) presented their experimental data on AoR and HR to show that both decrease as particle size increases. Cain (2002) observed a decrease in flowability rating with increasing mean particle size. The flowability rating was based on a correlation equation involving HR, mean particle size and AoR. Both the HR and the Carr index (CI) derived from compressibility studies were well received by industry due to the relative ease of their determination and excellent reproducibility. Materials with HR < 1.25 are considered to be non-cohesive while powders with HR > 1.4 are cohesive. Powders with HR in the range of 1.25–1.4 are in the transitional group (Iqbal & Fitzpatrick, 2006; Kamath et al., 1993; McGlinchey, 2005). It was found by some researchers that the correlations between HR, CI, and other measurements are rather poor (Bell, 2001; Soh, Liew, & Heng, 2006). Furthermore, measurement of compressibility is not always related to the flow and cohesiveness of materials. Tapped density in HR measurements is sensitive to the type of apparatus and the total number of taps applied (Abdullah & Geldart, 1999). Based on more extensive research, Geldart et al. (2009) showed that flowability measurements from aerated and non-aerated powders corresponded well with each other.

The Sauter mean size of each mixture was calculated from:

$$d_{sv} = \frac{1}{\sum_{i=1}^{n} x_i / d_{vi}}$$

Table 1

<table>
<thead>
<tr>
<th>Particle size, $d_p$ (µm)</th>
<th>Fine silica gel</th>
<th>Coarse silica gel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aerated density (g/cm³)</td>
<td>0.264</td>
<td>0.477</td>
</tr>
<tr>
<td>Tapped density (g/cm³)</td>
<td>0.449</td>
<td>0.531</td>
</tr>
<tr>
<td>Particle density, $\rho_p$ (g/cm³) (Pycnometer)</td>
<td>2.1292</td>
<td>2.1290</td>
</tr>
<tr>
<td>Hausner ratio (dimensionless)</td>
<td>1.7</td>
<td>1.1</td>
</tr>
<tr>
<td>Angle of repose (°)</td>
<td>46</td>
<td>31</td>
</tr>
</tbody>
</table>

Table 2

<table>
<thead>
<tr>
<th>Particle size, $d_p$ (µm)</th>
<th>Fine ballotini</th>
<th>Coarse ballotini</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aerated density (kg/m³)</td>
<td>0.808</td>
<td>1.420</td>
</tr>
<tr>
<td>Tapped density (g/cm³)</td>
<td>1.391</td>
<td>1.600</td>
</tr>
<tr>
<td>Particle density, $\rho_p$ (g/cm³) (Pycnometer)</td>
<td>2.4527</td>
<td>2.4836</td>
</tr>
<tr>
<td>Hausner ratio (dimensionless)</td>
<td>1.72</td>
<td>1.13</td>
</tr>
<tr>
<td>Angle of repose (°)</td>
<td>47</td>
<td>28</td>
</tr>
</tbody>
</table>
2.3. Methodology

2.3.1. Angle of repose (AoR)

Hosokawa PT-S was used to measure static AoR from a heap carefully built up by dropping the powder through a vibrating sieve and glass funnel onto a horizontal plate where a charge-coupled device camera (CCD) was used to capture the heap picture. The picture was then simulated using a software called PT-R emulation into AoR value as shown in Fig. 2.

2.3.2. Hausner ratio (HR)

The ratio between tapped density (defined as number of taps) (TD) and aerated bulk density (ABD), known as the Hausner ratio (HR), is often used as an internal friction index in cohesive powders (Iqbal & Fitzpatrick, 2006; McGlinchey, 2005). Steps explained in detail by Abdullah and Geldart (1999) were implemented to conduct the ABD and TD using the Hosokawa PT-S with 100 cm³ cup capacity and 180 as the number of taps for the tapped density. The excess powder was scraped from the top of the cup, without disturbing the loosely settled powder before weighing the sample to record the aerated density. The average of five tests was used on each sample for aerated density and tapped density.

2.3.3. Cohesion (S) using WSUMCT

As described in Section 2.2.1, WSUMCT is used to measure the cohesion strength (S) of the compacted powder. Moreover in the new set-up, and in view of the difficulties in getting enough volume of sample for testing, especially for expensive powders, the vaned paddle and Perspex sample cell are replaced in order to reduce the amount of powder required for each test. The new vane is 22 mm in inner diameter, 64 mm in outer diameter and 10 mm in height, as shown in Fig. 3, and the sample cell required 208 mL of sample, with an inner diameter of 94 mm and a height of 30 mm.

Sections 2.2.1 and 2.3.3 highlighted the two main strategies used in the modification of WSCT:

(i) To improve the accuracy with which the cohesion strength can be measured.
(ii) To reduce the volume of the sample required by 60%.

Fig. 4 displays a general profile of the torque vs. time, showing that the failure point of the powder has a sharp peak. The calibration curve (Orband & Geldart, 1997) to measure the torque was eliminated using WSUMCT. A compacted powder was prepared from each mixture by applying a load of 5 kg on the powder for 1 min before inserting the paddle. The cohesion value S was calculated.
using the following formula (Orband & Geldart, 1997):

\[ S = \frac{3T}{2\pi (R_o^3 - R_i^3)} \]  

where \( S \) is the cohesion strength (kPa) of the powder, \( T \) is the measured torque (mNm), and \( R_o \) and \( R_i \) are the outer and inner radii (cm) of the vane respectively.

3. Results and discussion

3.1. Powder SEM scan

A scanning electron microscope (SEM) [Philip XL 30 by Philips Holland] was used to obtain digital images of fine and coarse silica gel and also of fine and coarse ballotini powder samples at higher magnifications. Fig. 5 shows the images obtained between 500× and 5000× magnifications, for fine and coarse ballotini, spherical in shape, and for silica gel, porous and irregular in shape. Such irregularity causes considerable interlocking of particles and increases resistance to flow.

3.2. Determination of flowability

Fig. 6 shows that AoR as measured by the Hosokawa PT-S for the silica gel and ballotini powder mixtures decreases as the particle size increases, much in line with the findings of other researchers. The same trend is observed for the measurements of HR (Fig. 7) and cohesion strength \( S \) (Fig. 8).

According to Cain (2002), a static angle of repose greater than 40° indicates a cohesive powder, while an angle less than 40°, a non-cohesive powder, and by considering AoR 40° as a boundary angle between free-flowing and cohesive powders. Fig. 6 shows that accordingly a mean particle size of 34 \( \mu \)m will be the critical size for both the porous silica gel and the non-porous ballotini. The free-flowing behavior for a powder becomes cohesive when the mean particle size is less than this critical size. Heap

Fig. 5. SEM micrographs: (a) fine silica gel, (b) coarse silica gel, (c) fine ballotini and (d) coarse ballotini.
formation depends strongly on the interparticle forces which provide the strength to form the mechanical network while the momentum of falling particles tends to disrupt heap formation (Wong, 2000). The smoothness and roundness of the ballotini surfaces could greatly reduce the interparticle forces; therefore the momentum impact of falling for non-porous ballotini (especially for large particles) is greater than silica gel powder, enough to disturb heap formation and keep the AoR value low. Decreasing the amount of the large size particles forms a sudden change in the correlation slope, as shown in Fig. 6, when the mean particle size is less than 34 μm. Moreover for smaller particles the falling momentum is less. The disruption of the falling particles for silica gel is less than that for ballotini due to higher interparticle forces for silica gel.

Fig. 7 shows a linear relationship between HR and particle size for silica gel, indicating that 28 and 36 μm are the boundaries for the powder to behave like Group AC based on Geldart grouping, which corresponds to 1.25 < HR < 1.4, while the powder is free-flowing when the Sauter mean size is larger than 36 μm. Powder less than 28 μm in mean size is cohesive powder.

In contrast to silica gel powder, ballotini mixtures formed non-linear relationship as can be seen in Fig. 7. The mixture shows cohesive behavior when the Sauter mean size is smaller than 32 μm which corresponds to HR = 1.4, while it is free-flowing when the particle is larger than 38 μm. The fine powder will have smaller aerated density than the coarse powder because of its low density, and also the fine particles will stick to the wall causing higher voidage at the aerated state. During tapping, the fine powder will have more chances to compact more than the coarse powders, to result in higher HR.

The cohesion strength for the porous and non-porous was measured using WSUMCT, as shown in Fig. 8. WSUMCT is capable of detecting even small changes in cohesion value because of the applied torque (via the electrical motor) is measured by a very sensitive digital sensor, up to four decimal digits of N m torque, whereas this is not possible for the original WSBCT. In fact, there is yet no specific cohesion strength standard formula to indicate the flowability characteristics, in which the weighted cohesion as a new indicator for the powder cohesivity is established in this work as demonstrated in Section 3.3. Linear lines are obtained which proves that the measured cohesion is corresponding well to the general understanding of the cohesivity property for powders.

The linear relationship observed between AoR and cohesion S (Fig. 9) proves that S is a good flowability indicator. By applying the 40° criterion in Fig. 9, 0.075 and 0.172 kPa will be the boundary values for silica gel and ballotini mixtures respectively for cohesive and non-cohesive powders. Referring again to Fig. 8, 34 and 38 μm will be the corresponding cohesivity boundaries for silica gel and ballotini powders respectively. In other words the mixtures will be free-flowing when the particle mean sizes are larger than the observed critical values and they are cohesive when the mean particle sizes are less than these points.

By employing the 1.25 < HR < 1.4 criterion in Fig. 10 and dropping the cohesion values obtained in Fig. 8, the corresponding particle sizes are 29 and 41 μm for silica gel, implying that the powder is cohesive for mean size smaller than 29 μm and free-flowing for larger than 41 μm. For ballotini, the corresponding boundaries will be 30 and 43 μm. In Fig. 10, linearity is not possible to
Table 3  Calculated WS_A values for silica gel powders.

<table>
<thead>
<tr>
<th>d_v (μm)</th>
<th>AoR</th>
<th>HR</th>
<th>CI</th>
<th>ABD × 10^3 (kg/m³)</th>
<th>S (kPa)</th>
<th>WS_A × 10^3 (m²/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.4</td>
<td>48.0</td>
<td>1.65</td>
<td>21</td>
<td>0.269</td>
<td>0.191</td>
<td>0.710</td>
</tr>
<tr>
<td>19.0</td>
<td>45.4</td>
<td>1.56</td>
<td>21</td>
<td>0.405</td>
<td>0.172</td>
<td>0.425</td>
</tr>
<tr>
<td>23.0</td>
<td>42.4</td>
<td>1.45</td>
<td>19.14</td>
<td>0.459</td>
<td>0.050</td>
<td>0.108</td>
</tr>
<tr>
<td>25.6</td>
<td>41.5</td>
<td>1.41</td>
<td>18.34</td>
<td>0.45</td>
<td>0.043</td>
<td>0.096</td>
</tr>
<tr>
<td>33.2</td>
<td>39.4</td>
<td>1.25</td>
<td>15</td>
<td>0.43</td>
<td>0.113</td>
<td>0.266</td>
</tr>
<tr>
<td>39.0</td>
<td>39.2</td>
<td>1.21</td>
<td>10.8</td>
<td>0.44</td>
<td>0.026</td>
<td>0.055</td>
</tr>
<tr>
<td>43.6</td>
<td>37.4</td>
<td>1.14</td>
<td>7.63</td>
<td>0.47</td>
<td>0.000</td>
<td>0.000</td>
</tr>
</tbody>
</table>

Table 4  Calculated WS_A values for ballotini powders.

<table>
<thead>
<tr>
<th>d_v (μm)</th>
<th>AoR</th>
<th>HR</th>
<th>CI</th>
<th>ABD × 10^3 (kg/m³)</th>
<th>S (kPa)</th>
<th>WS_A × 10^3 (m²/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>28.0</td>
<td>43.0</td>
<td>1.53</td>
<td>18</td>
<td>0.909</td>
<td>0.186</td>
<td>0.205</td>
</tr>
<tr>
<td>32.3</td>
<td>39.0</td>
<td>1.34</td>
<td>15.3</td>
<td>1.298</td>
<td>0.180</td>
<td>0.139</td>
</tr>
<tr>
<td>38.1</td>
<td>37.4</td>
<td>1.24</td>
<td>14</td>
<td>1.391</td>
<td>0.169</td>
<td>0.122</td>
</tr>
<tr>
<td>46.5</td>
<td>37.0</td>
<td>1.22</td>
<td>10</td>
<td>1.422</td>
<td>0.160</td>
<td>0.11</td>
</tr>
<tr>
<td>52.2</td>
<td>36.0</td>
<td>1.19</td>
<td>9.5</td>
<td>1.454</td>
<td>0.155</td>
<td>0.107</td>
</tr>
<tr>
<td>59.6</td>
<td>35.0</td>
<td>1.20</td>
<td>7.8</td>
<td>1.291</td>
<td>0.140</td>
<td>0.108</td>
</tr>
<tr>
<td>69.4</td>
<td>33.0</td>
<td>1.13</td>
<td>7</td>
<td>1.422</td>
<td>0.115</td>
<td>0.081</td>
</tr>
<tr>
<td>72.0</td>
<td>28.0</td>
<td>1.13</td>
<td>6.6</td>
<td>1.42</td>
<td>0.093</td>
<td>0.066</td>
</tr>
</tbody>
</table>

cover all the data points for ballotini, suggesting that this powder performs differently for the different methods used. The fact of different classification techniques providing incompatible results is not exclusive in this work (Bell, Ennis, Grygo, Scholten, & Schenkel, 1994; Krantz, Zhang, & Zhu, 2009; Taylor, Ginsburg, Hickey, & Gheyas, 2000). However, this implies that aerated properties of ballotini powder cannot be applied to examine the non-aerated state properties.

3.3 Weighted cohesion indicator (WS_A)

The ratio between the measured cohesion (S) using WSUMCT to aerated bulk density (ABD) is named weighted cohesion (WS_A), as shown below:

\[ WS_A = \frac{S}{ABD} \]  

Calculation of WS_A is listed in Tables 3 and 4 for silica gel and ballotini powders, respectively. The transition limits for the powder behavior were highlighted according to the 40° AoR and HR theories.

Fig. 11 plots WS_A vs. AoR for the two powders used to produce a critical WS_A value at the intersection between the two correlation lines for the two powders used to reflect the 40° -theory at WS_A of 0.16 × 10^3 m²/s or 41° AoR with 98% accuracy. The WS_A value varies according to the type of the method originally implemented while it provides indication of the relative flow characteristics. Based on the WS_A vs. HR plots presented in Fig. 12, the lower and upper transition limits of WS_A are 0.11 × 10^3 and 0.16 × 10^3 m²/s, respectively. In other words, the powder is free-flowing for WS_A smaller than 0.11 × 10^3 m²/s. Drawing the dashed lines at HR = 1.25 and 1.4, shows that the two powders tend to overlap each other while the transition limits fall between these values.

The data observed above are summarized in Tables 5 and 6.

4. Conclusions

This paper describes a new flowability indicator named weighted cohesion (WS) measured by the Warren Spring-University of Malaya cohesion tester (WSUMCT), a modified Warren Spring-Bradford cohesion tester (WSBCT). WSUMCT exhibits the merit of requiring reduced sample amounts and being more accurate and operator-insensitive compared with WSBCT. The validation of WS tested by WSUMCT was authenticated by comparing with established measured Hausner ratio (HR) and angle of repose (AoR) for two groups of particle mixtures, porous and nonporous, showing that WS may provide a powerful technique to study directly the particle internal cohesion forces and use these data to indicate the powder flowability.
References