Physics International 1 (1): 16-21, 2010
ISSN 1948-9803
© 2010 Science Publications

Cohesiveness and Flowability Properties of Silica Gel Powder

E.C. Abdullah, A.M. Salam and A.R. Aziz
Department of Chemical Engineering, Faculty of Engineering,
University of Malaya, 50603 Kuala Lumpur

Abstract: Problem statement: The measurement of powder flowability is a major concern for most industrial processes that deal with the handling of bulk solids as raw materials, intermediates, or products. **Approach:** The influence of particle size on the flowability of silica gel was investigated using aerated and non-aerated methods. The aerated method involved the measurement of the Hausner Ratio (HR) and Angle Of Repose (AOR) using a Hosokawa Powder Tester (PT-S), while the non-aerated method involved a Cohesion Index (CI) measurement based on a Stable Micro Systems TA.XT Plus texture analyzer. The mean size diameters of the fine and coarse powders used in this work were 9.4 and 60 µm, with densities of 2.1262 and 2.1290 gm cm^{-3}, respectively. The experiments were conducted immediately after drying with a range of sizes of the chosen powder. **Results:** The three measured parameters confirmed the general expectation that the fine powders are cohesive and the coarse powders are free-flowing. The HR values indicated that the mixture was a free-flowing one above the 40 µm size. A transition in flowability occurred when the size was between 28 and 40 µm, while the mixture became cohesive when the particle size was below 28 µm. All three of the flowability indicators corresponded well with each other, signifying that the selected indicators correctly predicted the flowability. **Conclusion:** The new non-aerated method, where CI was measured, was proven to be a reliable indicator in predicting flow characteristics.

Key words: Powder, flowability, Hausner ratio, particle size, angle of repose, cohesivity

INTRODUCTION

Powders account for more than 50% of the raw materials used in the chemical industry. The food industry also produces many different types of powders, such as starches, milk powders, and many other spray-dried products. In the pharmaceutical industry, many medicines are produced in tablet form by the compression of powders. The chemical industry is probably the largest user of powders, with the widespread use of catalyst powders, and many of the final chemical products, such as polymers, are delivered as powders.

Numerous operations in process industries involve powder handling, such as: Storage of powders in hoppers and bins, transportation of powders from storage to the processing area, grinding or milling of powders, mixing of different powders to produce a desired quality, compression of powders in moulds to obtain a preformed solid product, granulation of powders to obtain larger grains which can be more easily transported and processed, and aeration by blowing gas upwardly through a powder bed in order to improve the contact between the powder particles and the aerating gas.

Flowability is the ability of granular solids and powders to flow. Flow behavior is multidimensional in nature, and it depends on many physical characteristics. Flowability, in fact, is a consequence of the combination of the physical properties of material that influence flow, environmental conditions, and the equipment used for handling, storing, and processing these materials, however there is no significant relationship for the trying to related flowability (Fitzpatrick *et al*., 2004). The capability of predicting powder flowability is helpful for preventing production stoppages in all bulk solid handling. However, no single test can fully quantify the flowability of a given powder. Some of the factors that affect the flowability of bulk solids and powders include particle size, moisture content, humidity, flow agents, temperature, and pressure.

Particle size and the particle-size distribution both play significant roles in flowability as do other properties, such as bulk density, angle of repose, and compressibility of bulk solids. Even a small change in

Corresponding Author: E.C. Abdullah, Department of Chemical Engineering, Faculty of Engineering, University of Malaya, 50603 Kuala Lumpur Tel: +60-3-79675301 Fax: +60-3-79675319

particle size can cause significant alterations in the resulting flowability. A reduction in particle size often tends to decrease the flowability of a given granular material due to the increased surface area per unit mass (Fitzpatrick *et al*., 2004; Fitzpatrick and Ahrne, 2005). The finer the particle size and greater the range of particle sizes, the greater the cohesive strength, and the lower the flowability were (Iqbal and Fitzpatrick, 2006). A reduction in particle size also increases the contact area between the particles, thereby increasing the cohesive forces.

Erica *et al*. (2009), in a study of static Angle Of Repose (AOR) observed a decrease in the value of the flowability rating with an increase in mean particle size, indicating an increase in flowability. The flowability rating was based on a correlation equation involving the Hausner ratio, the mean particle size and the angle of repose. Prior to the study of (Erica *et al*., 2009; Carr, 1965); Wong, 2000; Seville *et al*., 2000) and others have successfully used HR as a powder-flowability indicator.

Geldart *et al.* (2006) have studied the flowability for two types of powder (spherical porous Fluid Cracking Catalyst and angular non-porous Aluminum oxide trihydrate) using AOR and Hausner Ratio (HR) methods and they concluded that HR decreases as the particle size increases, confirming that an increase in the particle size of a powder mixture is always accompanied by a decrease in cohesiveness. The same trends were observed for the angle of repose, which increased for both materials as the mean particle size decreased. Based on the linear relationship between them, the authors claimed that both AOR and HR parameters are good indicators of powder flowability (Abdullah and Geldart, 1999).

Virginie *et al.* (2008) have investigated the flowability and cohesiveness for a large variety of wheat powders by employing the Powder Rheometer (Stable Micro System), which measures the Cohesion Index (CI). The results demonstrated a large diversity in flowability and cohesive properties for the powders examined. The particle-size distribution of wheat particles at low moisture content showed a significant impact on the CI. Earlier, Charu *et al.* (2005) implemented the Powder Texture Analyzer (SMS) for predicting the flowability of drugs powder, while Freeman (2007) studied powder flowability using a Powder Rheometer (FT3/FT4) by recording the energy consumed during the blade traverse at different speeds and claimed the machine has potential to indicate the powders cohesivity. However, to date no research has been done to conclusively confirm that CI can be used as a flowability indicator.

In this study, aerated (HR, AOR) and non-aerated (CI) methods are employed to investigate the influence of particle size on flowability of a porous silica gel powder which has been selected as the case-study powder. The validity of CI as a flowability indicator was evaluated by comparing it to trends in HR and AOR.

MATERIALS AND METHODS

Material preparation: Mixtures of fine and coarse silica gel powder, of mean sizes 9.4 and 60 µm, respectively, were selected as a model for porous industrial powder hence providing a range of increasing particle size mixtures from 9.4 up to 60 µm as shown in Table 1.

Mixtures were prepared by mixing the powders with a Kenwood Mixer (KMC 560) for five min. The mixtures were subsequently dried at 90°C for 3 h before each test was conducted.

Methodologies:

Hausner ratio: The ratio between tapped (defined as a certain number of taps) and aerated bulk density is known as the Hausner ratio and it is often used as an internal friction index for cohesive powders (Geldart *et al*., 1984).

The standard steps were used to conduct the Aerated Bulk Density (ABD) and Tapped Density (TD) using the Hosokawa Powder Tester PT-S with 100 cm^3 cup capacity and 180 as the number of taps.

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. A vertical tapping was applied on the cup after the extension cap had been fixed on top of the cup and extra powder added. For each sample, the operation was continued up to 180 taps, whereupon a steady state was reached, indicating that the packing had been achieved; subsequently the powder was weighed to calculate the tapped density.

Table 1: Silica gel powder mixtures with percentages of the fine powder

Percentage of fine	Sauter diameter (µm)
0	60.0
1	56.9
2	54.2
5	47.3
7	43.6
10	39.0
15	33.2
20	28.9
25	25.6
30	22.9
40	19.0
100	9.40

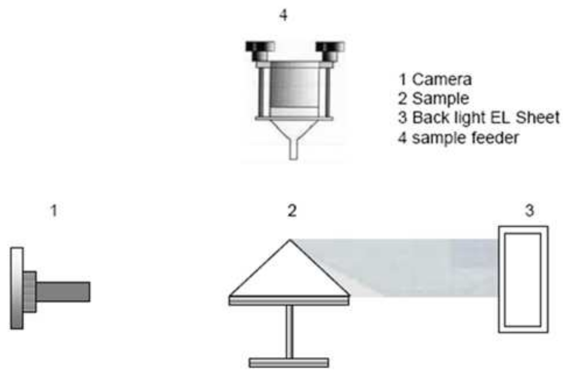

Fig. 1: Hosokawa CCD camera

A free-flowing powder has HR value less than 1.25 (Group A) and the cohesive powders have HR greater than 1.4 (Group C), while powders with HR values n the range of 1.25-1.4 are in the transition group (Group AC) (Abdullah and Geldart, 1999; Geldart *et al*., 1984). Five tests were conducted on each sample for aerated density and tapped density and the results averaged.

Angle of repose: Angle Of Repose (AOR) was measured from a powder heap carefully built up by dropping the powder through a vibrating sieve and glass funnel onto a horizontal plate using the Hosokawa PT-S. A Charge-Coupled Device camera (CCD) was used to capture an image of the heap profile and calculate the angle of repose value using a PT-R emulation, as shown in Fig. 1. From the established relation between the powder flowability and AOR, an AOR below 30° indicates good flowability, 30-45° some cohesiveness, and 45-55° very cohesive (Carr, 1965), while Geldart *et al.* (2006) suggested the use of a 40° criterion in classifying free-flowing and cohesive powders; this latter value was also adopted for this study.

Cohesion index: Cohesion Index (CI), which is defined as the ratio "Cohesion Coefficient/sample weight", was measured by using a Stable Micro Systems TA.XT Plus texture analyzer. The cohesion coefficient (g, mm) is the work required to lift the blade up through the powder column during the decompression phase at the speed of 50 mm sec^{-1}, which is determined by integrating the negative areas under the force-displacement curve in Fig. 2:

$$\text{CI}\ (\text{mm}) = \frac{\text{Cohesion Coefficient}\ (\text{g, mm})}{\text{Sample weight}\ (\text{g})}$$

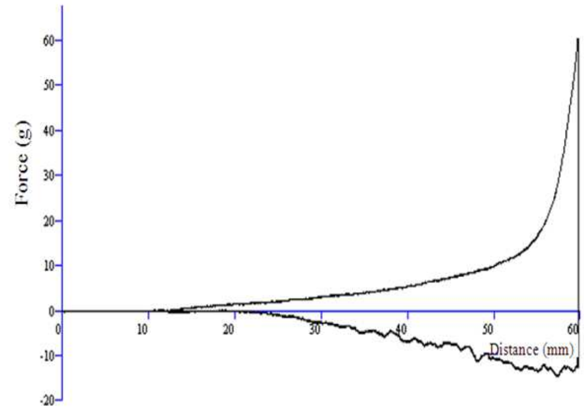

Fig. 2: General force-displacement

Fig. 3: Stable micro system TA-XT plus (SMS)

A sample of 30 cm^3 of each mixture (based on the formulations in Table 1) were prepared for each testing using a cylindrical vessel with a capacity of 60 cm^3 and set on the platform of the texture analyzer. A specified rotating blade (48 mm in diameter and 10 mm in height), which was able to move up and down, was rotated clockwise and anti-clockwise (Fig. 3).

The flowability properties were evaluated from the displacement during a controlled rotation of the blade inside the vessel (Virginie *et al*., 2008; Mukherjee and Bhattacharya, 2006).

Firstly, two conditioning cycles were performed to remove any stress history from the powder and to normalize the powder column after filling. The rotating blade was then moved down through the powder column using a "cutting" action to minimize compaction. The upward part of the cycle lifted the powder and the force of the powder on the vessel base was recorded.

Table 2: Classification of Cohesion Index (CI)

Cohesive Index (CI)	Flow behavior
>19	Hardened/extremely cohesive
19-16	Very cohesive
16-14	Cohesive
14-11	Easy-flowing
<11	Free-flowing

As mentioned previously, a low cohesion index is associated with non-cohesive free-flowing powders, while a high cohesion index is associated with cohesive, poorly flowing powders, as shown in Table 2. In addition to measuring CI, the SMS is also capable of predicting caking strength, compaction coefficient and speed-flow stability. However, these parameters are related more to powder cake formation and stability of the flow during the processing than to flow rate changes in powder handling. As the main purpose of this work was to study the effect of the particle size on the flowability, the discussion of these three parameters was left to a future publication.

RESULTS

It is observed from Fig. 4-6, for AOR, HR and CI, respectively, that smaller particles are more cohesive, while the cohesivity was diminished for larger particles.

By applying the 40° AOR criterion as the border between cohesive and free-flowing powder, powders less than 34 µm can be considered to be cohesive and vice-verse, as shown in Fig. 4. However, the universally accepted standard of particles less than 45 µm being considered a fine powder.

As these powders were not uniformly sized, being composed of complex distributions of sizes and shapes, the 34 µm value observed in Fig. 4 is acceptable.

The more frequently used parameter, HR, displayed a similar trend, as shown in Fig. 5. Two distinctive sizes were observed as bordering criteria: HR<1.25 and HR>1.4 as cohesive and free-flowing powders, respectively (Abdullah and Geldart, 1999; Geldart *et al.*, 1984). Silica gel of less than 28 µm diameter is cohesive and that larger than 36 µm is free-flowing (Fig. 5). The silica gel in-between 28 and 36 µm is in the transition group, which exhibits mixed cohesive and free-flowing behavior. Based on Geldart's powder grouping, the silica gel powder used here can be categorized as Group C for powders less than 28 µm, Group A for powders over 36 µm and Group AC for powders in-between 28 and 36 µm. CI has not yet been an established as a criterion in distinguishing cohesive and free-flowing powders, except for the criterion proposed by Stable Micro Systems Ltd., UK, as shown in Table 2.

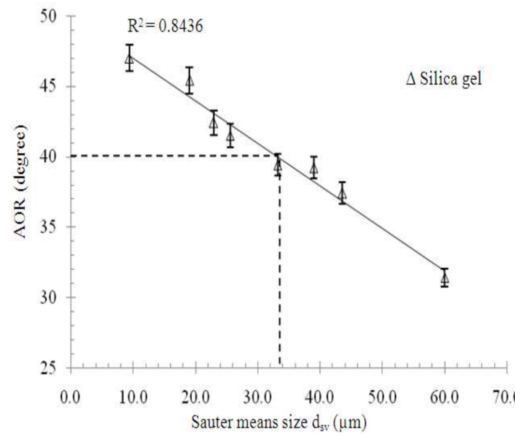

Fig. 4: Measured angle of repose for tested powers

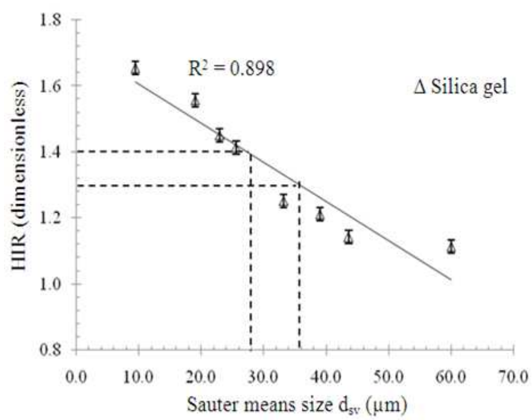

Fig. 5: Sauter means size effect on measured Hausner ration

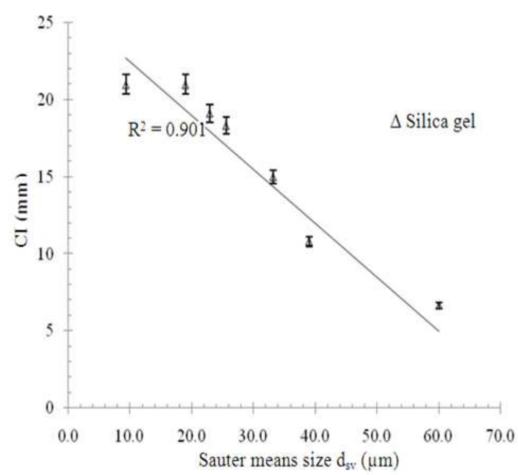

Fig. 6: Cohesion index linear correlation for tested powders with Sauter mean size

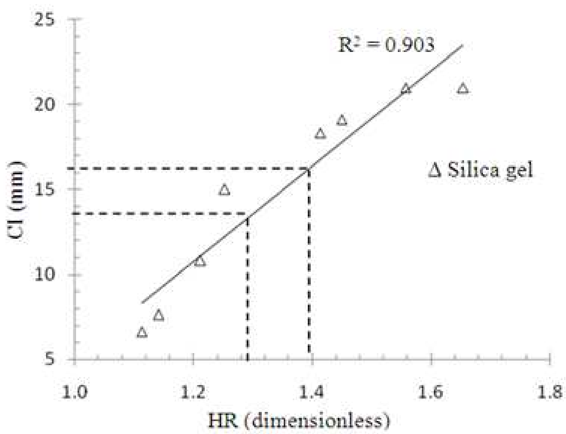

Fig. 7: Cohesion index and Hausner ration linear relationship trend

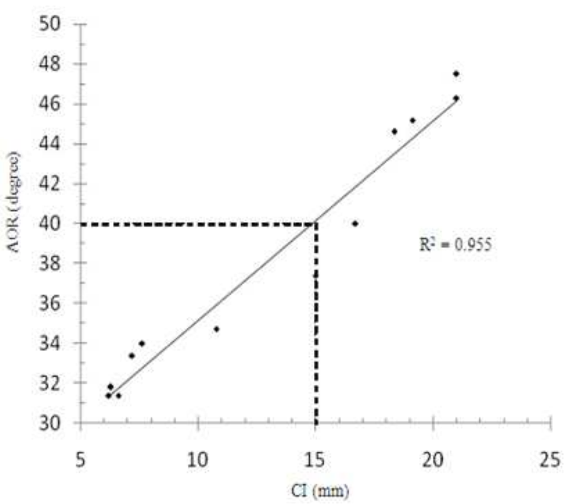

Fig. 8: Angle of repose Vs cohesion index

The classification proposed in Table 2 was based on in-house SMS research, but has not been thoroughly tested by other researchers. However, the trend shown in Fig. 6 corresponds well with the expected theory, as shown in Fig. 4 and 5.

The measured CI variation with Sauter means size is reported in Fig. 6 as a linear trend. These results reveal one main point: CI as indicator is sensitive enough to detect the powder size influence on the measured applied torque resistance by giving general profile where CI increases with particle size decreased.

Figure 7 displayed a linear relationship for obtained HR and CI while the linear relationship correlation between AOR and CI is presented in Fig. 8. Both trends authenticate CI as new flowability indicator as will be covered in the discussion.

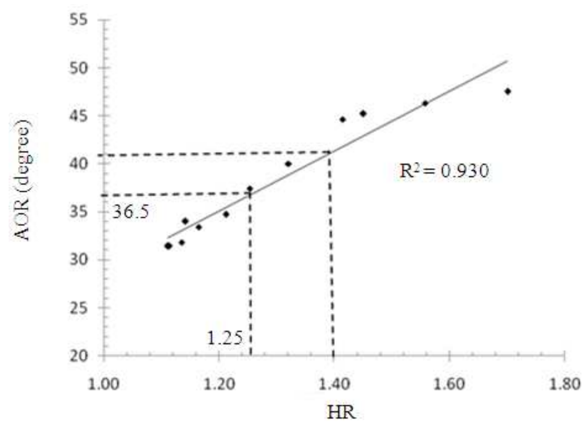

Fig. 9: Angle of repose Vs Hausner ration

DISCUSSION

The comparison of CI with HR indicates, a linear relationship which confirms that both indicators are providing powder flowability pointing in the same direction (Fig. 7). Based on the HR criterion, it can be established that CI>16 mm should indicate cohesiveness and CI<14 mm should indicate free-flowing. These two values are in exact agreement with the criterion proposed by Stable Micro Systems Ltd. Co.

Applying these values to Fig. 6, it can be concluded that powders <28 μm are cohesive and powders >35 μm are free-flowing. Furthermore, the transition group falls into the range of $28<d_p<35$ μm. This range indeed closely matches the range established from Fig. 5 and 6, which validates the cohesivity evaluation of the powder used. Referring to Fig. 8, by using the 40° criterion as the boundary between Groups A and C, it was found that the corresponding CI is 15 mm. Here again, referring to Fig. 6, the critical size for distinguishing between the two groups was 31 μm, which is close to the value established in Fig. 4.

Figure 9 just reconfirms that the two established powder flowability indicators (HR and AOR) provided good indication of the cohesively of the powders.

CONCLUSION

The impact of silica gel particle size on powder flowability was studied. The measured parameters clearly showed the effects of particle size on the powder cohesivity. The new non-aerated method, where CI was measured, was proven to be a reliable indicator in predicting flow characteristics. Furthermore, human interference is almost completely eliminated with this method. The proposed CI classifications, as shown in Table 2, corresponded perfectly with the presented results. The results also reconfirmed that HR and AOR are indeed good flowability indicators in the aerated method category.

REFERENCES

Abdullah, E.C. and D. Geldart, 1999. The use of bulk density measurements as flowability indicators. Powder Technol., 102: 151-165. DOI: 10.1016/S0032-5910(98)00208-3

Carr, R.L., 1965. Evaluating flow properties of solids. Chem. Engineer., 72: 163-169. DOI: 10.1080/02726350902775988

Charu, V.N., M. Shahrzad and F. Reza, 2005. Application of Powder rheometer to determine powder flow properties and lubrication efficiency of pharmaceutical particulate systems. AAPS. Pharm. Sci. Technol., 6: 49. DOI: 10.1208/pt060349

Erica E., J. Oliver, T. Pugsley, J. Sharma and J. Zhou, 2009. Flowability of moist pharmaceutical powders. Powder Technol., 189: 409-415. DOI: 10.1016/j.powtec.2008.06.017

Fitzpatrick, J.J. and L. Ahrne, 2005. Food powder handling and processing: Industry problems, knowledge barriers and research opportunities. Chem. Eng. Proc., 44: 209-214. DOI: 10.1016/j.cep.2004.03.014

Fitzpatrick, J.J., S.A Barringer and T. Iqbal, 2004. Flow property measurement of food powders and sensitivity of Jenike's hopper design methodology to the measured values. J. Food Eng., 61: 399-405. DOI: 10.1016/S0260-8774(03)00147-X

Freeman, R., 2007. Measuring the flow properties of consolidated, conditioned and aerated powders-A comparative study using a powder rheometer and a rotational shear cell. Powder Technol., 174: 25-33. DOI: 10.1016/j.powtec.2006.10.016

Geldart, D., E.C. Abdullah, A. Hassan Pour, L.C. Nwoke and I. Wouters, 2006. Characterization of powder flowability using measurement of angle of repose. China Particuol., 4: 104-107. DOI: 10.1016/S1672-2515(07)60247-4

Geldart, D., N. Harnby and A.C. Wong, 1984. Fluidization of cohesive powder. Powder Technol., 37: 25-37. DOI: 10.1016/0032-5910(84)80003-0

Iqbal, T. and J.J. Fitzpatrick, 2006. Effect of storage conditions on the wall friction characteristics of three food powders. J. Food Eng., 72: 273-280. DOI: 10.1016/j.jfoodeng.2004.12.007

Mukherjee, S. and S. Bhattacharya, 2006. Characterization of agglomeration process as a function of moisture content using a model food powder. J. Texture Stud., 37: 35-48. DOI: 10.1111/j.1745-4603.2006.00037.x

Seville, J.P.K., C.D. Willett and P.C. Knight, 2000. Interparticle forces in fluidization. Rev. Powder Technol., 113: 261-268. DOI: 10.1016/S0032-5910(00)00309-0

Virginie, L., C. Denis, M. Marie-Helene and C. Bernard, 2008. Flowability, cohesive and granulation properties of wheat powders. J. Food Eng., 86: 178-193. DOI: 10.1016/j.jfoodeng.2007.09.022

Wong, A.C.Y., 2000. Characterization of the flowability of glass beads by bulk densities ratio. Chem. Eng. Sci., 55: 3855-3859. DOI: 10.1016/S0009-2509(00)00048-8