A Review: Recent Progress on Evaluation of Flowability and Floodability of Powder†

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Abstract

Recent studies evaluating the flowability and floodability of cohesive powder under conditions of consolidation, mechanical force, vibrating force, fluid force, and floodability are reviewed. The ball indentation test is an effective method for evaluating the flowability of a small amount of cohesive powder at very low stress under consolidation conditions. The environmental conditions such as temperature and humidity play an important role in the flow of cohesive powder. With regard to cohesive powder flowing under mechanical force, the FT4 powder rheometer can evaluate the powder flowability using the total energy which is related to the shear stress on the impeller blade. The vibrating capillary method and the vibrating shear tube method are effective for the measurement of the flowability of strongly cohesive powder. The test using powder discharge by air flow can assess the flowability of cohesive powder which Carr’s flowability index is an equivalent level by using mass flow rate and interstitial air pressure. Finally, the flushing of cohesive powder occurs when the interstitial air pressure and the void fraction are high. The pressure difference between the interstitial air pressure and the outside of an orifice are the dominant factors in the spouting of powder.

Keywords: ball indentation, caking, rheometer, vibration, air pressure, flushing

1. Introduction

Powders and bulk solids are widely used in various industries such as material production, food processing and pharmaceutical development. When using these powders, they will go through several handling processes such as storage, discharge, transportation, mixing, filling, milling and separation. Recently, cohesive powder used in these processes is substantially increasing because it has many advantages, for instance, having a superior reaction potential due to the large specific surface area (Naito M. and Makino H., 2011). On the other hand, the handling of cohesive powder in air becomes extremely difficult because of cohesive forces arising from van der Waals force, electrostatic force and liquid bridge force. Cohesive forces also become stronger relative to gravity force as the particle size decreases (SPTJ, 2006). Therefore, adhesion and cohesion forces give rise to problems with powder handling equipment such as the stable operation of powder handling processes and the quality control of product material. Additionally, rat hole and bridge would occur upon discharge from storage and pipeline transportation if cohesive forces are strong. Unexpected discharge of cohesive powder from a small gap would also occur when cohesive powder is released from a hopper exit. These phenomena are known to cause trouble in the powder handling equipment and it is necessary to avoid them.

The typical flowing form of powder in equipment is classified by gravity flow, compression flow, mechanical flow, vibrating flow and fluid flow. This classification arises from the dynamic behaviour and it is deeply linked with the dynamic properties of powder flow. Furthermore, cohesive powder flow is influenced by the particle properties, for instance, size and shape, and by external factors such as external forces and the surrounding environment. As mentioned above, the understanding of cohesive powder flow is strongly connected with the operation of powder handling equipment. It is therefore necessary to evaluate flowability and floodability of powder in conditions as close as possible to the actual behaviour and operating conditions.

Carr (1965a, 1965b, 1969) suggested powder flow could be evaluated by using some powder properties. According to this method, the flowability is evaluated through angle of repose, compressibility, angle of spoutula, and cohesiveness or uniformity, whilst the floodability is evaluated through by angle of fall, angle of difference, dispersibility, and flowability index. In general, Carr’s method may...
be validated under static condition of bulk solids. However, this method is not suitable for the dynamic condition of powder because the evaluation of flowability and floodability is carried out using the above mentioned parameters. Therefore, a direct evaluation of flowability and floodability of powders is necessary, and there are many instruments for this purpose. Based on these backgrounds, this review introduces recent works for the evaluation of flowability and floodability by the classification of powder flowing in compressed flow, mechanical forced flow, vibrating flow, fluid flow, and flooding. It focuses on the dynamic measurement, flow mechanism, and the critical point of powder flow because they are important factors in the understanding of powder flow.

2. Powder flow under consolidation

Powders are often subjected to compressive forces in scenarios such as discharge to storage, compression moulding and tableting shoes. In these processes, the understanding of the yield strength is important for equipment design. If the powder has experienced normal and shear stresses from an external force then it would be possible to carry out an evaluation based on a shear test and consolidation test. With regard to the evaluation, Schwedes (2003) reported that there are many testers to measure the flowability of powders such as the Jenike shear cell (Jenike, 1967), Schulze ring shear tester (Schulze, 2008) and others. Amongst these testers, the shear cell is the most commonly used and there are many studies and reviews. Therefore, a detailed explanation for evaluation of flowability by shear cells is not given in this article. On the other hand, these testers need to improve the evaluation of the bulk cohesion and the tensile strength caused by indirect measurements. Some researchers pointed out the tensile strength is determined by extrapolating yield locus on the tensile region (Schulze, 2008), and is overestimated (Vasilenko et al., 2013). Zafar et al. (2015) mentioned the measurement of flowability of cohesive powders at low stress is difficult because the inter-particle contact forces have a strong effect. For these reasons, there is a request for a direct measurement of tensile strength and an evaluation method that can measure at a low stress level. In this section, the review focuses on new tests for the evaluation of flowability of cohesive powder under low stress conditions.

Hassanpour and Ghadiri (2007) proposed the ball indentation method on a powder bed. This method can be performed on small amounts of loosely compacted powders. They examined the flowability of bulk powders using an indentation test and an unconfined compression test, where a small assembly of powders was compacted at low pressure levels. Sample powders of about 5 g were formed as weak tablets, and they were consolidated into a cylinder by piston. The given piston pressure was from 3 to 20 kPa and the loading speed was 1 mm/min. The formed tablet was indented by using a spherical indenter of a glass sphere of 1.588 mm in diameter. Fig. 1 shows how the hardness calculation is carried out. The hardness of consolidated powder $H_D$ is calculated by the penetration depth and load during a test cycle.

$$ H_D = \frac{F_{\text{max}}}{A_i} \quad (1) $$

where $F_{\text{max}}$ is the maximum indentation load. $A_i$ is the projected area of the impression as follows:

$$ A_i = \pi (d_b h_c - h_i^2) \quad (2) $$

where $d_b$ is the diameter of indenter and $h_c$ is the critical indentation depth which is estimated through the interception of the tangent with the unloading curve as shown in Fig. 1.

Hassanpour and Ghadiri (2007) reported the yield stress obtained from indentation was similar to that of unconfined compression for the cases of $\alpha$-lactose and mag-
maximum carbonate, whilst the indentation results for the cohesive glass beads differed from those of unconfined compression. They speculated this difference might be attributed to constraint factor, which is affected by particle shape and inter-particle friction. Furthermore, through comparison between Distinct Element Method (DEM) simulation and experiments, they found a linear correlation between the unconfined yield stress and the consolidation pressure.

Wang et al. (2008) also experimentally investigated the indentation hardness of α-lactose monohydrate, micro-crystalline cellulose and starch powder. They found the constraint factor for a powder bed is not a single value, and concluded the constraint factor would depend on single particle properties such as particle shape, roughness and friction coefficient. Drawing on Hassanpour and Ghadiri (2007) and Wang et al. (2008), Pasha et al. (2013) carried out numerical analysis of the ball indentation method using DEM simulation. Their analysis results mainly revealed a minimum bed height of 20 particle diameters is required in order to achieve reliable hardness values, and small indenters with diameters smaller than 16 particle diameters exhibit fluctuations in hardness measurements. They also noted if the indenter size is too close to the die size, the walls provide confinement and increase the measured hardness.

Zafar et al. (2017a) analysed the operating conditions of the ball indentation method. In this study, five kinds of powders (three silanised cohesive spherical glass beads, limestone, and Durcal 15) were used. They showed a sieved method gave a uniform packing with minimum void fraction. They revealed the dimensionless penetration depth at a given consolidation pressure must exceed 0.4 and the minimum indenter size should be larger than 16 times the mean particle diameter as well as Pasha et al. (2013). The upper limit of the indenter to the bed diameter should be also smaller than 0.65. As a conclusion, they defined minimum indenter size, bed height, and bed volume as shown in Table 1.

Zafar et al. (2015) stated that it becomes difficult to obtain reliable values of the unconfined yield strength when the applied stresses were below 1 kPa. They experimentally examined the tensile strength and hardness of bulk cohesive powders by using the Raining bed method, the Sevilla powder tester, and the Ball indentation method, and compared them with the results obtained from the Schulze ring shear tester. From the relationship between tensile strength and applied stress, they reported the results of a shear cell and the Sevilla powder tester were in approximate agreement. They also found the measurement results of the hardness by the ball indentation method and the unconfined yield stress by the shear cell method increased linearly with the applied stress. This result shows clearly the hardness measurement of the powder bed by the ball indentation method is linked to the tensile strength. Based on these results, the ball indentation method can evaluate the flowability on a small quantity of cohesive powders at low stress levels.

In addition, the assessing of caking behaviour such as the consolidation flow of cohesive powders is important. Zafar et al. (2017b) undertook a comprehensive review on the mechanism and the testers of bulk powder caking. Recently, there have been new methods for measuring caking characterization, for example, the environmental caking tester (Calvert et al., 2013) and the Edinburgh powder tester (Thakur et al., 2014). Calvert et al. (2013) have introduced the environmental uniaxial caking tester which can control the temperature and relative humidity at the inlet and exit of the powder bed as shown in Fig. 2. The experiments were performed under 14 different conditions where the applied uniaxial stress, the temperature, the relative humidity and the consolidation time were varied. The sample powder had a mean particle diameter of 0.92 mm. They measured the stress and strain during the consolidation test and the maximum stress was taken as the cake strength. They found the cake strength increased with increasing consolidation stress, consolidation time, temperature and relative humidity. These results revealed the cake strength was related to the environmental and mechanical conditions to which the sample powder was exposed.

Thakur et al. (2014) have also studied the caking behaviour of spray-dried detergent powders using the Edinburgh powder tester. The tested powder had the same solid density and different moisture content and size ranges. They examined the relationship between unconfined strength and axial consolidation stress. As a result, a sample of higher moisture content displayed higher unconfined strength under the same conditions of consolidation stress. They surmised this was because the moisture increased the stickiness and plasticity of contact. A larger particle size also showed higher unconfined strength compared to a smaller one. Consequently, Thakur et al (2014) argued the coarse particles have higher moisture content and have more plastic deformation compared to small size.

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**Table 1** Minimum indenter and sample size requirement for ball indentation. Reprinted with permission from Ref. (Zafar et al., 2017a). Copyright: (2017) Elsevier B.V.

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<tr>
<th>Size $x_p$ (μm)</th>
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particles. They also pointed out the possibility of higher contact area and higher adhesion in case of the coarse particles.

Chen et al. (2017) have experimentally investigated caking behaviour by adopting the ball indentation method. In this study, they examined the effect of relative humidity, temperature and time against the hardness of the powder bed. The sample powders used were three pharmaceutical excipient powders (PVP, HPC and CaHPO₄) with different hygroscopicity. They discovered that due to the coupled effect of the increase in relative humidity and temperature on powder caking, the hardness of all the powders increased. They also found the glass transition of PVP appeared when the relative humidity was the higher value. This means that the state of the powder bed of PVP changed from “bonded” to “transformed” on adsorption of moisture. Furthermore, when the relative humidity of HPC changed, the samples transformed from a glassy state into a rubbery state, and then the particles fused together and the volume of the powder bed decreased significantly. These results show the change of cohesive forces between particles depend on the surrounding environment, and that indicates the environmental conditions and the particle properties are important parameters for evaluating the consolidation flow.

3. Powder flow by mechanical force

There are many equipments that mix powders by mechanical force, such as mixers, screw feeders, and rotary valves. The flow of powder in the equipment is a dynamic phenomenon, and dynamic flowability tests have been developed: the rheological properties of the powder are evaluated by the measurement of the stirring torque of the impeller and the blade in the powder bed (Satoh et al., 1994 and Bruni et al., 2005). In recent years, the powder rheometer method has been applied to evaluate the dynamic behaviour of powder, and one of the well-known tester is the FT4 powder rheometer (Freeman, 2007). This method evaluates the powder flowability by a total energy \( E \), which is calculated with Eq. (3) using the measured force and torque on a rotating blade in the powder bed.

\[
E = \int_0^h \left( \frac{T}{R \tan \beta} + F \right) dh
\]

where \( T \) is the torque on the impeller, \( F \) is the vertical force on the base, \( R \) is the impeller radius, \( \beta \) is the helix angle and \( h \) is the penetration depth.

However, the understanding of the flow mechanism of the powder rheometer has been requested, and many researchers have experimentally and numerically examined the flowability of powder in the FT4 (Bharadwaj et al. 2010 and others).

Bharadwaj et al. (2010) have investigated the flow of non-cohesive particles in the FT4 using DEM simulation. They examined the effect of particle properties such as size, shape and friction by the simulated force and torque on the impeller blade in the particle bed. The particles used were spherical glass beads ranging from 1 to 3 mm, and the aspect ratio was also varied from 1.0 to 2.0. There were also glass beads of an irregular shape. They reported the particle size and size distribution have a minimal effect on the mixing torque and force, whilst the particle shape was an important factor because the force and torque increases for non-spherical particles as compared to spherical particles. Furthermore, they noted the sliding and rolling friction coefficients were also important factors to determine the force and torque on the blade.

Hare et al. (2015, 2017) have explored the flow mechanism of cohesive powder in the powder rheometer. They carried out direct measurements of DEM parameters such as the surface energy and the elastic and plastic stiffness, and analysed the dynamic behaviour of silanised cohesive glass beads ranging from 1.7 to 2.1 mm by DEM. Fig. 3 shows the relation of total flow energy and coefficient of
sliding friction, and they pointed out the flow energy receives the influence of the coefficient of sliding friction between particles. Fig. 4 shows a comparison with the flow energy calculated using the vertical forces acting on the base and the impeller. It shows that the flow energies using the force acting on the impeller and the base were almost same. They mentioned the resistance to flow was predominantly rotational, i.e. $T/R \tan \beta >> F$.

Furthermore, the stresses in front of the blade were also calculated by the DEM. They indicated both average compressive stress and deviatoric stress increased almost linearly with the blade penetration depth. Therefore, they suggested a characteristic shear stress can be determined for a powder under a given test condition in the FT4.

Wilkinson et al. (2017) have examined the mixing and segregation in the FT4 using DEM and varied five input parameters (the static and rolling friction coefficients, coefficient of restitution, Young’s modulus, and the cohesion energy density). They confirmed the findings of Bharadwaj et al. (2010) and Hare et al. (2015, 2017), and found the static and rolling friction parameters play a critical role in the determination of the basic flow energy (BFE) and the specific energy (SE) of the powder bed. In addition, the cohesion energy density between particles also played a significant role in the change of BFE. This means that the cohesiveness of the powder will influence the flowability of the powder.

Yan et al. (2016) have examined the mixing and segregation in the FT4 by using DEM simulation. They used the binary mixtures with the different particle size ratio and volume fraction. To evaluate mixing and segregation during the blade movement, the number of contact particles in the mixture of two particles were evaluated by the segregation index. They found the segregation index and the flow energy decreased with increasing the number of test cycles in case of layered particles. On the other hand, when the initial filling state was mixed, the segregation index and the flow energy increased. Furthermore, a large particle size ratio led to a higher segregation and flow energy rate. They also showed the size ratio and the volume fraction were influenced by the change of flow energy gradient. These results suggested the FT4 could compare and rank the segregation tendency for particulate materials.

Pantaleev et al. (2017) have also performed an analysis on powder mixing in the paddle blade mixer by using DEM. They used the measurement result of flow energy in the FT4 for the optimization of the model parameters in DEM simulation. The powder used was the zeolite powder with cohesiveness which varied depending on the moisture content and the bulk density. The measurement result by the standard BFE test in the FT4 showed the flow energy increased markedly when the moisture content had a high value. To this result, they argued this trend was related to the significant contribution of inter-particle cohesive forces such as liquid bridging. However, the difference in flow energy was not clear at the range with the intermediate moisture content powders. From these results, it might be necessary to improve the sensitivity of the measurements in the FT4.

Nan et al. (2017) have analysed the dynamic behaviour of the non-cohesive particle bed under aeration conditions in the FT4 by using both experiments and numerical simulations. They carried out the standard downward test and the permeability test. The particles used were polyethylene particles and the simulations were performed in the same geometry as the FT4 experiments. They simulated the flow energy, the strain rate and the stresses when the tip speed and the superficial gas velocity were varied. As a result, they found both flow energy and shear stress increased linearly with increasing strain rate and also decreased with the increase in gas velocity. Furthermore,
they indicated the relationship between the flow energy and the shear stress was clearly correlated. This result suggested the shear stress could determine the flow energy in the FT4 as also outlined by Hare et al. (2015, 2017).

Nagashima and Ishikura (2015) have experimentally examined the influences of blade speed, consolidation and aeration on the total energy in the dynamic test of the FT4. They also measured the compressibility in the bulk test which carried out the volume reduction by applying a normal stress to the sample powder. In the experiment, ten kinds of powders which belonged to the classification of Geldart A, B and C were used. They showed the total energy could be evaluated by the compressibility and the cohesiveness when the blade speed and the gas flow were changed. Furthermore, the difference in total energy of the Geldart A, B and C particles clearly varied. In the aerated tests with the Geldart C particle, they observed agglomeration of particles and channelling into the powder bed. This result indicated the Geldart A and B particles were easily fluidized by aeration test and the total energy could evaluate the flowability of powder of the different particle sizes. Additionally, Bruni et al. (2007a, b) and Tomasetta et al. (2012) have also examined the rheological properties under aeration and fluidization by using a mechanically stirred fluid-bed rheometer.

Leturia et al. (2014) focused on the effect of the state of compaction and void fraction on bulk solids for the evaluation of powder flowability. They carried out the experiment of the rotational shear cell test, the consolidation test, the dynamic test and the aeration test by using the FT4, and also performed a tap density volumeter and the fluidization tests. In this study, five materials from nanoparticles to group B particle were selected and two binary mixtures were also used. They arranged the relationship between the flow index of the shear cell measurements and the compressibility of the consolidation tests under packed bed conditions. This result indicated the powder flow can be divided in three main regions such as easy/free flowing powders, cohesive powders, and intermediate behaviour. They also compared the Hausner ratio measurements with the flow rate index (FRI) of the dynamic test under the free surface conditions. From this result, the powder flow can be classified in two regions and the dynamic test was a quick way to evaluate flow properties of free flowing to relatively cohesive powders. Furthermore, under the aeration conditions, the aeration test was also a quick way to evaluate the effect of aeration on powder flow properties. This method can be used to evaluate if a powder is likely to be fluidized. On the other hand, they pointed out a more detailed understanding of the physical phenomena on the powder rheometer measurements is still needed, and the connection of several characterization methods is required to ensure a complete understanding of the powder flow properties over a wide range of conditions.

Recently, Li et al. (2018) have performed the standard dynamic test by using the FT4, where cohesive powders such as three kinds of CaCO₃ and ZrO₂ were used. They compared the results of BFE and FRI, and pointed out these values were difficult to distinguish between very similar materials. Therefore, they aimed to develop a new dynamic testing method which was based on the autocorrelation analysis of the torque measurements on the FT4. In the experiment, six powders belonging to Geldart group A, B and C were tested. They observed the powder behaviour during testing, and analysed the raw data of normal force and torque measurements. The results of cohesive powders found an empty space based on the particle agglomeration appeared behind the blade, and the torque oscillations such as a periodic behaviour occurred. They argued this oscillation was significant in the cohesive powder compared to a free-flowing powder, and an autocorrelation analysis was applied to evaluate the flowability of powder. The results showed the oscillation amplitude and the cycle time increased with increasing the powder cohesiveness. On the other hand, a free-flowing powder exhibited a liquid-like behaviour and showed almost no periodic motion. From these studies of the FT4, this method could be available to evaluate the flowability of powders, whilst it seems to need the improvement of the evaluating flowability of cohesive powders.

4. Powder flow under vibration force

With regard to the powder handling by using the vibration field, there are a number of options such as feeder, drying, and powder blocking prevention. These operations induce complex powder flow which depends on vibration amplitude and frequency. It is therefore important to understand the mechanism of powder flow in this field. In general, the feeding of smaller particles becomes difficult because of the effect of their cohesiveness, and intermittent powder flow will occur (Matsusaka et al., 1995a, 1996). These phenomena are because fine particles will agglomerate by the effect of cohesiveness. Therefore, in order to achieve reliable fine powder handling, vibration is applied to cohesive powder and flowability of powders under a vibrating field is an important subject.

Matsusaka et al. (1995a, 1996) have developed microfeeding of fine powders by using a vibrating capillary tube. Their powder feeder consists of a powder discharge hopper, a glass tube, a capillary tube, where the inside diameter of the capillary tube ranges from 0.4 to 1.6 mm. This feeder had horizontal vibration which was smaller than 760 Hz in frequency. In the experiment, three alumina powders and a fly ash powder were used where the
size range was from 6 to 20 μm. They revealed the discharged mass from the vibrating capillary tube for all the powder can be strictly controlled with this system. Fig. 5 shows the schematic particle flow near the inside wall under the horizontal vibration. It was found only the particles near the inside wall of the capillary tube were flowing randomly by the effect of cohesive forces, and the other particles inside the tube were transported downwards as a lump of powder. In addition, they indicated the critical condition of the powder discharge was related to the vibration acceleration, and a high frequency will be necessary to discharge the cohesive powder. Furthermore, Matsusaka et al. (1995b) have achieved the discharge of fine powder as small as 0.4 μm by using a capillary tube along with ultrasonic vibration applied by using a piezoelectric transducer of 20 kHz. They observed fine powder passed through the vibrating capillary tube, and the powder movement was approximated by plug flow with a thin boundary layer near the inner wall. They also found the continuous discharge for fine powder by using this method was possible.

Building on the former results, Jiang et al. (2006) have proposed the evaluation method of flowability of powder based on the vibrating capillary method. Jiang et al. (2009) have explored the performance of this system, in which a capillary tube was vibrated horizontally by a piezoelectric vibrator. The range of sample powders was from 5.2 to 58.2 μm. They examined the dynamic powder flowability by the relationship between the mass flow rate of powder and the vibration acceleration. According to their result, when the vibration acceleration exceeded a critical value, the powder flow began. The critical vibration acceleration was determined as the value where the powder flow rate exceeds 2 mg/s. They also observed the voids in the particle layer at a capillary tube by the CT images, and found the packing structure distribution transitioned from inhomogeneous to homogeneous when the vibration acceleration increased. Furthermore, Jiang et al. (2009) analysed the relationship between the critical vibration acceleration and the angle of repose to evaluate the powder flowability. The result found both the critical vibration acceleration and the angle of repose decreased with increasing mass median diameter. Furthermore, the variation of the critical vibration amplitude was sufficiently larger than that of angle of repose. This result showed the vibrating capillary method can have a high resolution in the flowability analysis.

Ishii et al. (2011a) have experimentally investigated the flowability of ZrO2 and WO3 particles by using the vibrating tube method. These particles had different concentrations, with mixtures made by coarse particles from 106 to 250 μm and fine particles under 45 μm. In this study, they evaluated the powder flowability by the two characteristic mass flow rates: the maximum mass flow rate for the increase in the vibration acceleration and the mass flow rate for the decrease in the vibration acceleration. They found the two characteristic mass flow rates were capable of evaluating the flowability because these experimental curves against the concentration of fine particles were similar to the results of Carr’s flowability index. Furthermore, they showed the relationship between the Carr’s flowability index and the two characteristic mass flow rates, and argued the sensitivity of the vibrating tube method was higher than that of the Carr’s flowability index. Additionally, Ishii et al. (2011b) have challenged to reduce the sample amount required for the measurement and evaluation of flowability. As a result, they concluded the flowability can be measured using 10 g or less of particles, and the vibrating tube method is suitable for measuring the flowability of a small amount of particles.

In light of the results reviewed above, measurement of the flowability by vibrating capillary tube turns out to be a simple method, because the powder being discharged only from a capillary. However, if the adhesion forces between particles are too large, bridging will occur in the capillary, hence the measurement of powder discharge will be difficult even though the vibration is increased. To solve the problem of the capillary method, Zainuddin et al. (2012) have developed an evaluation method for powder flowability by using the vibrating shear tube method. In this system, the powder can be discharged through a narrow gap between a vibrating tube edge and a flat bottom surface, since the particles receive high shear forces to overcome the adhesion and friction forces. Fig. 6 summarizes the schematic mechanism of powder flow in the vibration shear tube method. To evaluate the static properties of the powder flowability, they proposed the critical vibration acceleration which was determined by a threshold of mass flow rate at the beginning of particle discharge. On the other hand, to evaluate the dynamic properties related to the dynamic friction of particles, a
characteristic mass flow rate at a vibration acceleration of 300 m/s² was introduced, because a stable powder flow was observed for all the samples on this acceleration. As a result, they found the flowability depends on the mass median diameter, and high vibration was required to disintegrate the agglomerates into small pieces, and to flow through the gap. Furthermore, they stated that with this method, an improvement of the measurement efficiency can be expected by reducing the measurement time and sample amount.

Horio et al. (2014) have also examined the effect of particle shape on the powder flowability by using the vibrating shear tube method. In this research, microcrystalline cellulose particles were used, and the variation of the aspect ratio was from 1.8 to 6.4. The critical acceleration and the characteristic mass flow rate were determined in the same way as Zainuddin et al. (2012). They showed the angle of repose and the compressibility of powders were related to the critical acceleration and the characteristic mass flow rate. Furthermore, they indicated higher aspect ratios resulted in higher static and dynamic friction forces. This means the vibrating shear tube method can detect the effect of particle aspect ratio in powder flowability. From these results, the vibrating capillary method and the vibrating shear tube method can be recognized effective in the evaluation of flowability of the cohesive powders.

5. Powder flow by fluid force

With respect to powder flow by fluid force, the fluidization and aeration of powder are widely used in operations such as a reaction, drying, and transportation. This is because good gas-solid contacts and heat transfer characteristics can be obtained. Furthermore, the blow tanks and the air slides are classified as the powder supply equipment of the fluidization type. In these apparatuses, the fluidized air is applied into the powder, and the influences of inter-particle force and particle-wall surface force are reduced. Based on this operation, the flowability of powder is improved, and a smooth supply of the powder is achieved. In general operation of fluidization and aeration, the permeability air is supplied through the gas distributor at the bottom of the powder bed in a vessel. Geldart (1973) explored that the group A and B particles can be fluidized easily by permeated air, and the group D particle is possible to fluidize by large inertia force. On the other hand, it mentioned the fluidization of the group C particle was extremely difficult, because of the influence of an increased cohesion force. Therefore, the evaluation of cohesive powder flow using fluidization and aeration operations is highly important. To this issue, the powder discharge from an orifice by fluidization and aeration is well known, and there are so many researches such as the examples below.

De Jong et al. (1969/70, 1975a, b) have investigated the discharge characteristics of the group B particle from an orifice at the bottom of a vessel, where air was supplied from the bottom of the powder bed or both the bottom and the top of the bed. They proposed an empirical correlation of the mass flow rate \( m_p \) as a function of pressure difference between powder bed and outside of orifice \( p_{se} \), particle density \( \rho_p \), initial void fraction \( \epsilon_i \), orifice diameter \( D \) and particle diameter \( x_p \):

\[
m_p = C_d \frac{\pi}{4} (D - k_p x_p)^2 \sqrt{2(1 - \epsilon_i)} \rho_p p_s
\]

where \( C_d \) is discharge coefficient and \( k_p \) is particle shape factor.

Meanwhile they referred the supply position of air was not important. Geldart and Haesebrouck (1983) also experimentally examined the spouting of powder from an orifice at the bottom of the powder bed by using the groups A, B, C and D particles. They compared between the experimental results and a correlation as well as De Jong (1969/70). Furthermore, Martin and Davidson (1983) carried out the discharge experiments of the group A particle from the side of the vessel. The result revealed the mass flow rate of powder was proportional to the square root of pressure difference as the results of previous researchers.

On the other hand, when fluidization and aeration are applied to cohesive powder such as the group A and C particles in Geldart classification, the control of powder flow would be difficult. This means the cohesive forces between particles (such as van der Waals force, electrostatic force, and liquid bridge force) and the particle-fluid interaction forces have a strong influence on the powder flow. Therefore, the evaluation of flow for cohesive powder under the same operating conditions is important. Re-
Regarding this issue, Donsi et al. (2003) and Barletta et al. (2007) have investigated the discharge properties of powder from an orifice by using air of aeration and fluidization, which was introduced at the bottom of the powder bed of the group C particle (such as magnesium carbonate, silica, etc.). According to their results, the fluidization of the cohesive powder in the vessel by giving air flow was possible, whilst large agglomerates and cracks occurred in the powder bed. They also observed the aggregate diameter of discharged powder from an orifice was in the range from 1 to 6 mm. These results can be understood the group C particles couldn’t maintain the primary particle size by the influence in cohesive forces. Donsi et al. (2004) also reported the rat hole and funnel flows were arising when the given air velocity was lower. Meanwhile, they found the mass flow phenomenon of cohesive powder bed was exhibited when the air velocity was over the minimum fluidizing velocity. Furthermore, Cannavacciuolo et al. (2009) have challenged the fluidized discharge using cohesive powders of the range of micron and submicron sizes. They revealed a high air velocity was needed in order to eliminate the phenomena of piping and bridging of the powder bed. The result indicated the air velocity given to the powder bed was an important factor to suppress the unsteady flow of the cohesive powder.

Additionally, Ogata (2008, 2009) has experimentally examined the spouting phenomenon of cohesive powder bed through a circular orifice at the bottom of a vessel by using air flow. The powder used was a soft wheat flour of 46 μm with a particle density of 1493 kg/m³. This powder belongs to the group A particle, and the powder discharge from an orifice by gravity force was difficult. In this study, the air was supplied with three ways such as only the top of the powder bed, only the bottom, and both the top and bottom. As a result, when the same flow rate of air was simultaneously introduced both the top and bottom of the powder bed, the steady flow of cohesive powder was obtained. They also revealed critical conditions for the continuous spouting of the powder from an orifice existed. Ogata et al. (2013) have also attempted the discharge of silica powder which belongs to the group C particle. They found the flow pattern of the powder bed in a vessel became mass flow. They also referred the interstitial air pressure on the powder bed and the initial void fraction were strongly related with the beginning of the powder discharge and the mass flow rate of the powder. From the above results, the cohesive powder in group A and C particles can be smoothly discharged by using this method. However, these studies did not focus on the evaluation of flowability of cohesive powder.

Based on the previous studies, Ogata et al. (2015) have explored the evaluation of flowability of cohesive powder by using the test of the powder discharge from an orifice by air flow. The powders used were three kinds of Calcium hydroxide from 9.6 to 41 μm with the same particle density. In this study, they determined the beginning conditions of powder discharge by the relation of the continuous powder discharge through an orifice and the interstitial air pressure on the powder bed. They also explained the flow mechanism of cohesive powder using air flow as shown in Fig. 7. On the initial condition in Fig. 7(a), the free-flowing of cohesive powder through an orifice at the bottom of the powder bed was difficult to achieve because of the effect of cohesiveness between particles. When the air was supplied from both the top and the bottom of the powder bed, they observed the cohesive powder flow did not occur as shown in Fig. 7(b). This conditions were the low interstitial air pressure and the low initial void fraction.

On the other hand, as shown in Fig. 7(c), when the interstitial air pressure was increased, the cohesive powder was discharged through an orifice at the bottom of the powder bed. They concluded the difference in the powder flow was deeply linked to the relation between the cohe-

![Fig. 7 Schematic of mechanism of powder flow in the test of powder discharge by air flow. Reprinted with permission from Ref. (Ogata et al., 2015). Copyright: (2015) J. Soc. Powder Tech, Japan.](image-url)
sive force and the separation force on the particles. As for this flow mechanism, in order to start of powder flow, the separation force caused by the air pressure should overcome the cohesive forces on the powder in a stationary state.

Furthermore, Ogata et al. (2017) estimated the separation force on a particle in the powder bed based on Rumpf equation (Rumpf, 1970 and Tsubaki, 1984) as follows.

$$H = \frac{xp_x^2}{k_c(1-\epsilon_i)}$$ \hspace{1cm} (5)

where $H$ is the separation force, $p_x$ is the interstitial air pressure at the beginning of powder discharge, $x_p$ is the mean particle diameter, $\epsilon_i$ is the initial void fraction of the powder bed, and $k_c$ is the coordination number by Ridgway-Tarbuck correlation (1967).

They also expressed the cohesiveness on the powder by the ratio of separation force $H$ and gravity force $F_g$ as following equation.

$$\frac{H}{F_g} = \frac{6p_x}{\rho_g x_p(1-\epsilon_i)(13.8 - \sqrt{175 - 232(1-\epsilon_i)})}$$ \hspace{1cm} (6)

In addition, they evaluated the flowability of cohesive powder using the relationship between the mass flow rate of powder and the averaged interstitial air pressure at the top of the powder bed. In this study, three kinds of calcium hydroxide with different particle diameters were used. The mean particle diameter of an original powder, Ca(OH)$_2$-A, was 13.1 μm and other particle diameters, i.e. Ca(OH)$_2$-B and Ca(OH)$_2$-C, were 14.1 μm and 2.5 μm, respectively. Flowabilities of these powders using Carr’s method were in order: Ca(OH)$_2$-A = Ca(OH)$_2$-C < Ca(OH)$_2$-B.

As for experimental conditions, the initial void fraction and the interstitial air pressure were varied. Fig. 8 shows the relationship between the mass flow rate and the averaged interstitial air pressure under the natural packing. The result showed the mass flow rate of the smallest powder, Ca(OH)$_2$-C, was lower than the other two powders, and unsteady flow was arising. They also confirmed the mass flow rate of Ca(OH)$_2$-A decreased slightly compared to that of Ca(OH)$_2$-B. Based on these results, they revealed flowabilities of three powders were in order; Ca(OH)$_2$-C < Ca(OH)$_2$-A < Ca(OH)$_2$-B. Furthermore, as the result in Fig. 9, they found the cohesiveness of these powders was classified by Ca(OH)$_2$-B < Ca(OH)$_2$-A < Ca(OH)$_2$-C.
The flowability and the cohesiveness of the powder. In addition, they inferred a main factor of cohesive force in this study was van der Waals force. From these results, this method can be applied in the evaluation of the flowability and the cohesiveness of the powder.

6. Flooding phenomenon of powder

If the cohesive powder in a hopper contains a large amount of air, the powder flows out suddenly from a hopper exit. In general, this phenomenon is called the flooding or the flushing of powder, and can induce serious trouble in powder handling equipment such as rotary feeders, mono pumps, and silos. Therefore, the understanding of the flow mechanism of flooding is necessary. With regard to this subject, Schwedes (2003) explained flooding of powder as a fluid-like behaviour of fine particles which are fluidized by the gas flow. The author quoted the results of the flooding tendencies by Geldart and Williams (1985), and described the tendencies were classified by Geldart diagram (Geldart, 1973). As a result of the review, the flooding of the group D particles cannot occur, and the group B particles indicate only a low flooding tendency. Furthermore, the group A particles are easily fluidized and they have a remarkable flooding tendency. On the other hand, the group C particles are very cohesive, and their flooding tendency is lower. Additionally, author mentioned the flooding tendencies cannot be investigated in shear testers. Regarding the measurement of powder flooding, some studies are available. Carr (1965a, 1965b, 1969) proposed the prediction method of the floodability of powder. This method needs to measure four kinds of powder properties such as angle of fall, angle of difference, dispersibility and flowability index. However, this method could not predict occasionally an exact flooding phenomenon when a fine powder of the group C particle was used. It is therefore necessary to understand the flow mechanism of flooding.

Flooding phenomenon is deeply related to aeration and deaeration of the powder bed. Therefore, there are former studies based on this point. Bruff (1969) has focused on the aerated powder which induces the flooding, and proposed a simple tester as an indicator of the flooding. This study experimentally investigated the trend of flooding which was measured by the state of a forming pile of discharged powder from a glass tube. In the experiment, the powder was filled in a tube installed vertically, and was deaerated by using the vertical vibration. As the measurement, the shape of pile of powder on the flat plane was observed. It was concluded the flooding was arising when an angle of pile of powder was small. Lloyd et al. (1987) have also examined the deaeration of powder, and developed an annular shear cell of the Couette type to study in detail the flooding phenomenon for alumina powder. They found the shear stress was larger reduced when small quantities of air entrainment were introduced. They also revealed the transition from normal Coulomb-solid flow to liquid-like flow at high shear rates. Furthermore, their results suggested the addition of fine particles under 40 μm in diameter were greatly influencing the flooding of powder. Rathbone et al. (1987a, b) theoretically and experimentally analysed the aeration, the deaeration, and the flooding of fine powder, and referred the deaeration was a most effective factor for the prevention of flooding. However, the above researchers have focused merely on aeration and deaeration of powder bed. In a practical process, this phenomenon will induce an unstable mass flow rate and the powder leakage from an exit of the discharge equipment, and there is not an accurate evaluation method for the flooding phenomenon. On this subject, there are some works about the flow mechanism of the flushing by using an external loading, vibrating, and other forces.

Tomita et al. (1994) have explored the threshold conditions of the flushing generation. The tested powders were eight kinds of materials from 14 to 99 μm which belong to the group A and C particles. The experiment was conducted with a powder discharge device using a piston pressure at the top surface of the powder bed in a cylindrical vessel which has a small side orifice between 2 and 4 mm near the bottom. Fig. 10 shows an example of the flushing phenomenon, where an orifice diameter is 4 mm, piston pressure is 2.77 kPa and the initial void fraction is 0.73 (Kuchii, 1997). The powder used is calcium carbonate of 24.6 μm which has a particle density of 2690 kg/m³. This powder belongs to the boundary of the group A and C particles in the Geldart diagram. From the figure, the powder spouts out of an orifice due to the piston pressure acting on the surface of the powder bed, and this flow...
fraction is an important factor governing the flushing phenomenon. Geldart et al. (1985) have referred to the void fraction in the group A and C particles agreeing with the results of the Geldart diagram, and it was confirmed flushing occurred when the flowability index of the powder is larger than the floodability index. According to Carr’s method (1965a), flushing arises easily if the floodability index has a higher measured value. However, Kuchii et al. (1996) pointed out that powders having a high floodability index are not always occurring to flushing as shown in Fig. 11. Based on this results, they suggested the flushing occurs when the floodability index of the powder is larger than the flowability index, i.e. $FL \geq FW$. Furthermore, the results of the occurrence of flushing were plotted on the Geldart diagram, and it was confirmed flushing occurred in the group A and C particles agreeing with the result of Geldart et al. (1985). In addition, they referred the void fraction is an important factor governing the flushing phenomenon.

Furthermore, Kuchii and Tomita (2002) have defined the flushing phenomenon as follows; when an external load is applied to the powder, the spouting of powder is generated by an increase in interstitial gas pressure in the powder bed. They carried out the experiments by using a rectangular vessel which has a slit at the bottom. They also numerically simulated by gas-solid two-phase flow model using Darcy’s law. In this study, the tested particles were soft wheat flour and FCC catalyst which belong to the group A particle. They analysed the flow pattern during the flushing and the velocity distribution on the powder bed. The results showed the particle flow began near the slit at the bottom of vessel when the piston load was applied, and the velocity of particles is larger than that of free-flowing under gravitational acceleration. They also mentioned an increase in the interstitial fluid pressure appeared near the slit. This result indicated the interstitial air pressure greatly influences the flushing of powder.

Harada et al. (2002) have experimentally and numerically investigated the spouting of fine powder from a side orifice on a vessel by using the vertical vibration. The result showed both the intermittent powder flow and the continuous powder flow in response to vertical vibration. They found the powder spouted out like a plug when the vibration amplitude was comparatively small. Furthermore, when the vibration amplitude was large, they confirmed the powder flowed out like an intermittent dilute jet and dense flow. Furthermore, they also proposed the mechanism of the spouting of powder by vertical vibration as follows; (1) the relative motion of the powder bed to the vessel is induced by the vibration, (2) the pressure fluctuation is generated in response to the change in the gap between the bed and the vessel base, (3) the pressure difference between the vessel and the outside leads to periodic in- and out-flow of air through the orifice, and (4) the particles near the orifice spout out intermittently. Fig. 12 shows the schematic mechanism of motion of powder bed and percolation fluid by using the vertical vibration (Harada, 2004). Additionally, they examined the dependence of the mass flow rate on the vibration and the particle properties by using dimensional analysis. The results showed the mass flow rate of powder was in proportion to the generated air pressure and in inverse proportion to the vibration frequency. They concluded the generated air flow influences the spouting of powder.

Tashiro et al. (2006a) have explored the onset condition of flushing from an orifice at the bottom of a flushing vessel by using the impact force of free falling of the fluidized soft wheat flour, where the particle properties were a mean particle size of 69.8 μm, a particle density of 1417 kg/m³, and the moisture content between 11.9 and 13.4 %. The equipment consists of an upper hopper and a
flushing vessel which was arranged below an upper hopper as shown in Fig. 13. In the experiment, the mass flow rate of powder from an upper hopper was changed to give different impact forces on the powder in a flushing vessel. To measure the mass flow rate of flushing powder from a flushing vessel, the orifice diameter at the bottom of the flushing vessel was also varied. They showed the flushing occurs when the interstitial air pressure at the bottom of the flushing vessel was larger than 300 Pa, the initial void fraction was more than 0.54, and the mass flow rate of powder from an upper hopper exceeded 500 g/s.

Tashiro et al. (2006b) have also investigated the effect of the interstitial air pressure, the void fraction, the fluidized powder head and the orifice diameter of flushing vessel. In this study, a flushing vessel was directly fluidized, and the powder spouted out of an orifice at the bottom of the vessel as shown in Fig. 14. They found the interstitial air pressure at the bottom of the fluidized bed was proportional to the powder head, and its slope depended on the void fraction of powder. Furthermore, the liquid like characteristic was lost with the decrease in the void fraction of the powder bed. They also revealed the mass flow rate during the flushing was proportional to the square root of interstitial air pressure, and decreased with the orifice diameter.

From the above studies on the flushing phenomenon, it can be concluded that the interstitial fluid pressure and the void fraction greatly influence the flushing phenomenon, and the spouting of powder is dependent on the pressure difference between the interstitial air pressure and the outside of an orifice. Consequently, it can be understood the void fraction of the powder bed and the interstitial fluid pressure generated inside the bed are the dominant factors in the flushing of cohesive powder.

7. Conclusion

Recent progress in research on the evaluation of the flowability and floodability of cohesive powder has been reviewed. The contents have been classified by actual powder handling processes such as consolidation, mechanical force, vibration force, fluid force, and flooding. The ball indentation test is available as an effective method because it can assess the flowability of a small amount of cohesive powder at very low stress compared to shear cell. The environmental conditions such as temperature and humidity will influence the particle proper-
ties, and these parameters play an important role on the flow of cohesive power. It is therefore necessary to use the evaluating equipment of powder flowability accounting for these parameters such as the environmental caking tester.

FT4 powder rheometer can evaluate the powder flowability using the total energy which is estimated from the torque on the impeller and the vertical force on the base. From the results of investigation of flow mechanism in this tester, shear stress can determine the flow energy for a powder under given test conditions in the FT4. On the other hand, it is necessary to note the results are influenced by particle properties such as particle shape and cohesiveness.

Regarding the evaluation of flowability of cohesive powder using vibration, the vibrating capillary method and the vibrating shear tube method have been developed. These systems can evaluate the flowability by using the mass flow rate and the vibration acceleration. These are effective operations to evaluate the flowability of strongly cohesive powder.

Evaluation method of flowability using cohesive powder discharge from an orifice by air pressure has also been introduced. This method can evaluate flowability based on the relationship between mass flow rate of powder and average interstitial air pressure at the top of the powder bed. Additionally, cohesiveness of powder can be also studied by using the ratio of separation force and gravity force on a particle.

To understand the mechanism of flushing, several methods such as external loading, vertical vibration, and the fluidized powder flow have been introduced. The results revealed the flushing occurred for high interstitial air pressure and void fraction, and the spouting of powder from an orifice is strongly related with the pressure difference between the interstitial air pressure and the outside of an orifice.

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Nomenclature

- \( d_h \): diameter of indenter (m)
- \( D \): orifice diameter (m)
- \( E \): total energy (J)
- \( F \): vertical force acting on the base (N)
- \( F_g \): gravity force on a particle (N)
- \( F_{\text{max}} \): maximum indentation load (N)
- \( FL \): Carr’s floodability index (–)
- \( FW \): Carr’s flowability index (–)
- \( g \): gravitational acceleration (m s\(^{-2}\))
- \( h \): penetration depth (m)
- \( h_c \): critical indentation depth (m)
- \( h_t \): residual indentation depth (m)
- \( h_{\text{max}} \): maximum indentation depth (m)
- \( H \): separation force (N)
- \( H_D \): hardness of the consolidated powder (Pa)
- \( k_c \): coordination number (–)
- \( k_p \): particle shape factor (–)
- \( m_p \): mass flow rate (kg s\(^{-1}\))
- \( p \): pressure inside vessel (Pa)
- \( p_a \): pressure difference (Pa)
- \( P_{\text{avg}} \): average interstitial air pressure (Pa)
- \( p_o \): pressure at outside of an orifice (Pa)
- \( p_s \): interstitial air pressure at the beginning of powder discharge (Pa)
- \( R \): impeller radius (m)
- \( T \): torque acting on the impeller (N m)
- \( x_p \): mean particle diameter (m)
- \( \beta \): helix angle (degree)
- \( \rho_p \): particle density (kg m\(^{-3}\))
- \( \varepsilon_i \): initial void fraction (–)

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