Shear Testing of Powders for Process Optimization

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ABSTRACT

In the present paper the principle of shear testing is explained. Further, it is shown how the results of a shear test can be applied on the optimization of powder flow in a process, either by modification of the flow properties of the powder, or by equipment design based on the powder properties.

INTRODUCTION

Two groups of methods are used for the characterization of the flow behaviour of powders. The first group comprises phenomenological methods where a powder specimen is subjected to a particular procedure in order to deduce the flow properties from the observed behaviour (e.g., angle of repose, Hausner ratio)[1,2]. Possible drawbacks of these methods may be the device-dependency, and the influence of further physical effects[1].

The second group of methods uses a powder mechanics approach which is based on the work done by Andrew W. Jenike[3]. Jenike developed a theory describing the flow properties of a powder with well-defined physical quantities, and introduced the Jenike shear tester to measure these quantities.

BACKGROUND

Fig. 1 shows two cylinders with frictionless internal walls, one filled with a Newtonian liquid (a), the other filled with a powder. In the vertical direction a vertical force, F, is exerted on the liquid or the powder, respectively.

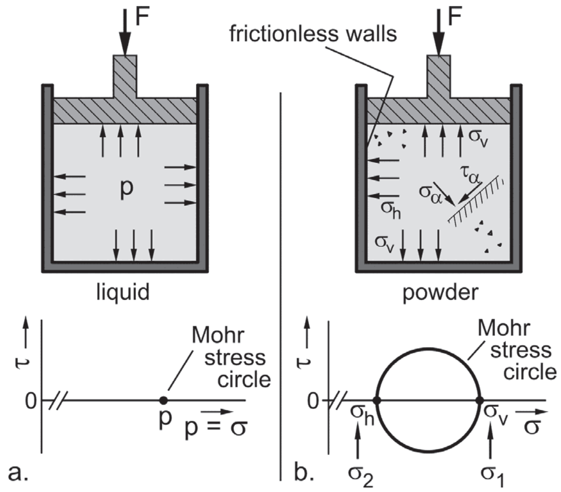

Figure 1. Newtonian liquid (a) and powder (b) compressed vertically.

In the Newtonian liquid (Fig. 1.a) the pressure, p, would be of equal magnitude independent of direction. Within the powder (Fig. 1.b) the horizontal stress, σ_h, is less than the vertical stress, σ_v, exerted on the powder from the top. The ratio of horizontal stress to vertical stress is called the stress ratio, K. Typical values of K are between 0.3 and 0.6[1].

It follows that in a powder different stresses can be found in different cutting planes. Thus, the behaviour of the powder is quite different from that of the liquid.

Stresses in cutting planes other than the vertical and the horizontal can be analyzed using a simple equilibrium of forces[1]. No shear stresses, τ, are exerted on the top or bottom surface of the solid element in Fig. 1.b, i.e., the shear stresses in horizontal planes are equal to zero. No shear stresses are acting at the frictionless lateral walls. Based on a simple equilibrium of forces on a volume element with triangular cross-section cut from the solid element shown in Fig. 1.b, the normal stress, σ_α, and the shear stress, τ_α, acting on a plane inclined by an arbitrary angle α, can be calculated[1].

The pairs of values (σ_α, τ_α) for all possible angles α form a circle when plotted in a σ-τ-diagram. This circle is called "the Mohr stress circle". The Mohr circle represents the stresses acting on all possible cutting planes within a solid element.

A Mohr stress circle intersects with the σ-axis at two points. The normal stresses defined by these points of intersection are called the principal stresses. The larger principal stress is designated as the major principal stress, σ_1, and the smaller as the minor principal stress, σ_2 (Fig. 1.b). Since in the example of Fig. 1.b no shear stress is acting on both the horizontal and the vertical planes ($\tau = 0$), the vertical stress, σ_v, which is greater than the horizontal stress, σ_h, is the major principal stress, σ_1.

An important qualitative result of the above analysis is that shear stresses can occur in powders at rest. This is impossible for a Newtonian liquid at rest. Therefore, a representation of the stresses (= fluids pressures) acting on different cutting planes of a Newtonian liquid at rest would yield a stress circle with the radius zero (Fig. 1.a).

Yield locus

If a powder does not flow in a specific situation, its strength is too high with respect to the stresses acting on it. The strength of a consolidated solid can be represented by a yield limit. In Fig. 2.a a specimen of a consolidated cohesive powder within two hollow cylinders is shown (mass $m = 0$, cross-sectional area A). The upper part of the specimen, while loaded by a vertical force, F_N, shall be moved horizontally by a horizontal force, F_S. The stresses, σ and τ, in the assumed shear plane (Fig. 2.a) at the underside of the upper part of the specimen, are a result of normal force, F_N, and shear force, F_S, i.e., $\sigma = F_N/A$, and $\tau = F_S/A$.

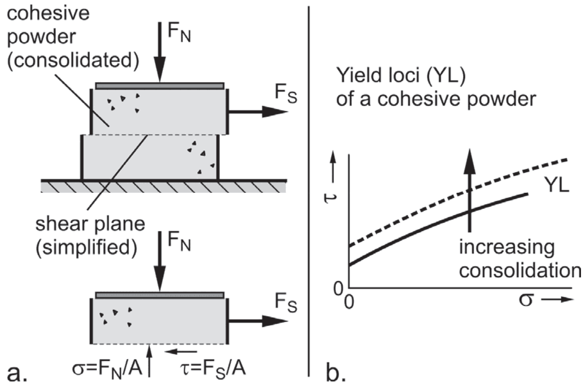

Figure 2. a. Measuring of the yield limit of a consolidated powder specimen, b. yield loci (yield limits).

To move the upper part along the shear plane, the friction within the powder must be overcome (inertia forces are neglected). For this F_S and, thus, τ must be sufficiently large. As known for Coulomb friction, the horizontal force, F_S, required to initiate movement is increasing with normal force, F_N. Therefore, in a σ-τ-diagram the shear stress is increasing with increasing normal stress thus forming an upward sloping curve, the yield limit (Fig. 2.b) which is called yield locus in powder technology. For the cohesive powder regarded here, the yield locus does not run through the origin because of the adhesive forces acting between the particles, i.e., even for a normal stress $\sigma = 0$ a shear stress $\tau > 0$ is needed. Yield loci of cohesive materials are often slightly curved whereas a yield locus of a free-flowing, non-cohesive powder would be a straight line running through the origin.

For different normal stresses, the yield limit represents the shear stress which is necessary to initiate movement (flow). With increasing consolidation, yield loci are shifted towards higher shear stresses (see dotted yield locus in Fig. 2.b).

Unconfined yield strength and flowability

Flowability of a particular powder can be determined quantitatively be measuring its strength. This is demonstrated by a uniaxial compression test (Fig. 3). A hollow cylinder with frictionless walls is filled with a powder specimen. The specimen is loaded vertically by the consolidation stress which is called σ_1 because it is identical to the major principal stress as explained in Fig. 1. Due to the consolidation load, both the density (bulk density) and the strength of the powder specimen increase.

After consolidation the hollow cylinder is removed. If subsequently the consolidated cylindrical specimen is loaded with an increasing vertical compressive stress, it will break (fail) at a certain stress, which is called the unconfined yield strength, σ_c.

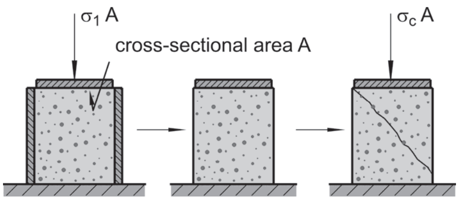

Figure 3. Uniaxial compression test.

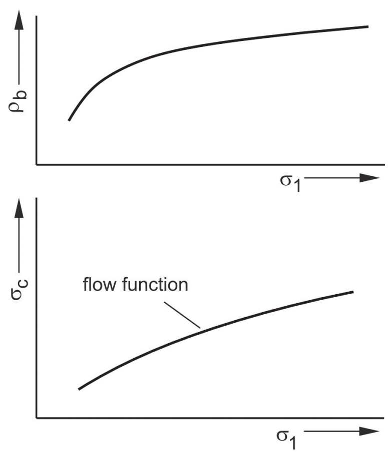

Figure 4. Bulk density, ρ_b, and unconfined yield strength, σ_c, vs. consolidation stress, σ_1.

Bulk density, ρ_b, and unconfined yield strength, σ_c, increase with increasing consolidation stress, σ_1 (Fig. 4). The function $\sigma_c(\sigma_1)$ is called flow function.

Since the strength of a powder must be overcome to initiate flow, flowability of a powder can be defined based on its unconfined yield strength, σ_c, in dependence on consolidation stress, σ_1:

$$ff_c = \sigma_1/\sigma_c, \quad (1)$$

The larger flowability ff_c, the easier a powder is set to flow. The following classification, based on a proposal by Jenike[3] gives an idea on the meaning of different values of ff_c:

$ff_c < 1$	not flowing
$1 < ff_c < 2$	very cohesive
$2 < ff_c < 4$	cohesive
$4 < ff_c < 10$	easy-flowing
$10 < ff_c$	free-flowing

ff_c depends on consolidation stress because σ_c is not proportional to σ_1 (Fig. 4). Thus, each value of ff_c as well as the corresponding classification is related to the stress level the powder is subjected to[1].

Some powders increase in strength if they are stored for a longer time at rest under a compressive stress (e.g., in a hopper). This effect is called time consolidation or caking. Time consolidation can be determined with the test shown in Fig. 3. For this the specimen is subjected to consolidation stress, σ_1, for a defined period of time (e.g., one day). Afterwards the unconfined yield strength is determined following the principle explained above (Fig. 3). Flowability is then calculated with Eq.(1).

Unconfined yield strength and yield locus

In Fig. 5 the uniaxial compression test of Fig. 3 is explained by combining stress circles and yield limit.

During consolidation the vertical normal stress, σ_1, acts on the top of the specimen.

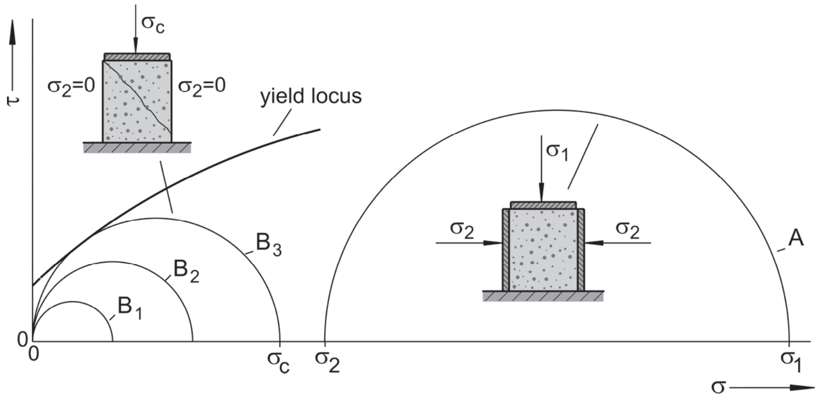

Figure 5. Measurement of unconfined yield strength.

Perpendicular to the vertical stress the (smaller) horizontal stress prevails according to stress ratio K (as in Fig. 1.b). The Mohr stress circle representing the stresses in this situation is plotted in the σ-τ-diagram shown in Fig. 5 (circle A).

In the second part of the test (Fig. 3), the specimen is loaded with increasing vertical stress while the horizontal stress is equal to zero (free lateral surface). Both vertical and horizontal stresses are principal stresses where the Mohr stress circle intersects with the σ-axis since in these planes no shear stress is acting. The stress states at different load steps are represented by stress circles with increasing diameter (stress circles B_1, B_2, B_3 in Fig. 5).

The specimen fails as soon as the Mohr stress circle touches the yield locus (circle B_3) and, thus, the yield criterion is met: In the cutting plane of the specimen, represented by the point where the Mohr stress circle touches the yield locus, the shear stress is high enough to move the particles across each other, according to the definition of a yield limit[1].

SHEAR TEST

With a shear test a yield locus is measured directly. Similar to the uniaxial compression test, the shear test is done in two steps: First the powder specimen is consolidated (preshear). Subsequently, a point of the yield locus is measured (shear to failure).

For preshear the powder specimen of cross-sectional area A (A seen from the top) is first subjected to a vertical normal stress, $\sigma = \sigma_{pre}$ (Fig. 6.a). Afterwards the top is moved horizontally (velocity v) relative to the fixed bottom in order to shear the specimen (Fig. 6.b). Due to friction between particles a shear stress, τ, is acting which is transferred to the top and measured (as shown in Fig. 7, left). With increasing shear stress the resultant force, F_R, acting on the specimen, increases. Thus, the specimen becomes increasingly consolidated, and the bulk density increases.

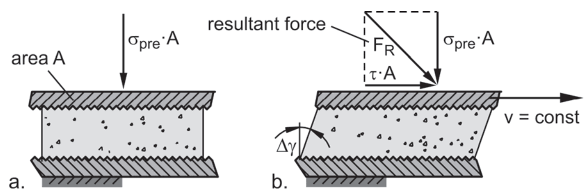

Figure 6. Powder specimen:
a. initial loading with normal stress σ;
b. shear deformation.

With time the slope of the shear stress vs. time curve decreases, and finally shear stress remains constant due to the friction within the powder being fully mobilized. The latter is called steady-state flow. From this point strength and bulk density do not increase further, and the specimen is

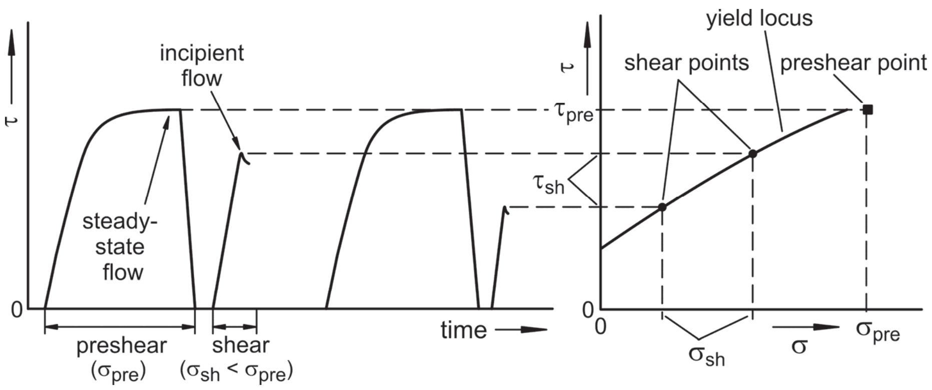

Figure 7. Plot of shear stress vs. time; yield locus.

"critically consolidated with respect to normal stress, σ_{pre}"[3]. The bulk density, ρ_b, and the shear stress, τ_{pre}, attained at steady-state flow are characteristic for the applied normal stress at preshear, σ_{pre}.

After consolidation by the preshear procedure, the shear stress is reduced to zero. The pair of values of normal stress and shear stress at steady-state flow (σ_{pre}, τ_{pre}) is plotted in σ-τ-diagram (Fig. 7, right). Point (σ_{pre}, τ_{pre}) is called the "preshear point".

For shear to failure the normal stress acting on the specimen is decreased to a value σ_{sh}, which is less than the normal stress at preshear, σ_{pre}. If the consolidated specimen is sheared under the normal stress $\sigma_{sh} < \sigma_{pre}$, it will start to flow (fail) when a sufficiently large shear stress is attained. At that point particles start to move against each other. After failure is attained, the material in the shear zone will start to dilate (decrease in bulk density), and shear resistance and, thus, shear stress will decrease. The maximum shear stress, τ_{sh}, characterizes incipient flow. The pair of values (σ_{sh}, τ_{sh}) is a point on the yield locus (shear point) of the consolidated specimen.

In order to measure the course of the yield locus, several of the tests described above are performed, where the specimens first must be consolidated at identical normal stress, σ_{pre} (preshear). Then the specimens are sheared to failure under different normal stresses, $\sigma_{sh} < \sigma_{pre}$. Each test yields the same preshear point (σ_{pre}, τ_{pre}), and one individual shear point (σ_{sh}, τ_{sh}) in accordance with the different normal stresses, σ_{sh}, applied at shear. The yield locus follows from a curve plotted through all measured shear points (Fig. 7, right).

Yield locus

The parameters describing the flow properties are determined from the yield locus (Fig. 8). The relevant consolidation stress, σ_1, is equal to the major principal stress of the Mohr stress circle which is tangential to the yield locus and intersects at the point of steady state flow (σ_{pre}, τ_{pre}). This stress circle represents the stresses in the sample at the end of the consolidation procedure (stresses at steady state flow). It is similar to the stress circle at the end of consolidation at the uniaxial compression test (stress circle A in Fig. 5). The unconfined yield strength, σ_c, results from the stress circle which is tangential to the yield locus and which runs through the origin (minor principal stress $\sigma_2 = 0$). This stress circle represents a similar stress state as the one which prevails in the second step of the uniaxial compression test (B_3, Fig. 5).

Knowing σ_1 and σ_c, flowability, ff_c, as defined in Eq. (1), is calculated. Thus, the most important quantity for the characterization of the flow behaviour of powders is known. However, for applications such as hopper design, which go beyond merely determining flowability, further parameters (flow properties) can be determined from the yield locus[1]. Bulk density, ρ_b, attained after consolidation, is determined from mass and volume of the specimen.

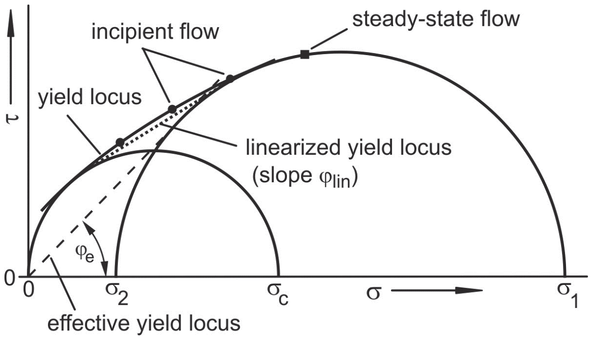

Figure 8. Yield locus, analogy to uniaxial compression test.

If several yield loci are measured at different stress levels, i.e., with different normal stresses at preshear, σ_{pre}, the above mentioned flow properties (e.g., unconfined yield strength, bulk density) can be indicated as a function of consolidation stress, σ_1, similar to Fig. 4.

RING SHEAR TESTER

Ring shear testers (rotational shear testers) have been used in soil mechanics since the 1930s[4]. In the 1960s Walker designed a ring shear tester for bulk solids[5], where lower stresses than in soil mechanics are of interest. In the following decades different ring shear testers have been built and investigated at several universities. In 1992 the author developed a manually operated ring shear tester[6,7], followed by computer-controlled versions for automatic shear testing, with the goal to enable easy operation and high accuracy.

Fig. 9 shows the principle of the shear cell of a small ring shear tester (series RST-XS)[8]. The ring-shaped (annular) bottom ring of the shear cell contains the powder specimen, and the annular lid is placed on top. The lid is fixed at a crossbeam.

A normal force, F_N, is exerted to the crossbeam and transmitted through the lid to the powder specimen in order to apply a normal stress σ to the specimen. To shear the powder, the lid and the bottom ring of the shear cell must rotate relative to each other. This is accomplished by rotating the bottom ring in direction of arrow ω (ω is the angular velocity), whereas the lid and the crossbeam are prevented from rotation by two tie-rods connected to the crossbeam. Each of the tie-rods is fixed at a load beam, so that the forces, F_1 and F_2, acting in the tie rods can be measured.

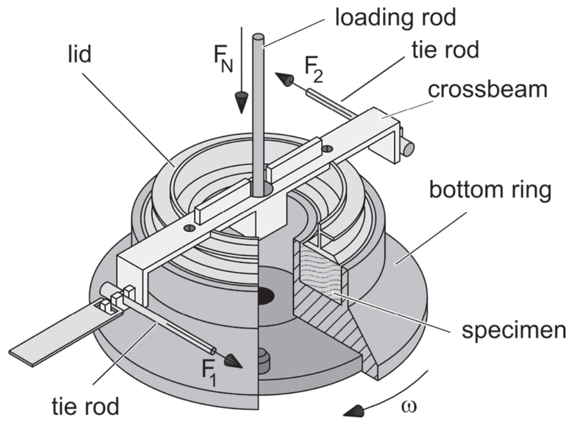

Figure 9. Shear cell of a ring shear tester (Schulze ring shear tester type RST-XS).

Bottom of the shear cell and lower side of the lid are rough in order to prevent the powder from sliding relative to these surfaces. Therefore, rotation of the bottom ring relative to the lid creates a shear deformation within the powder specimen. Through this shearing the powder is deformed and thus a shear stress τ prevails. Shear stress, τ, is calculated from forces F_1 and F_2. The specimen volume and bulk density, ρ_b, respectively, are determined from the vertical position of the lid and the mass of the specimen.

The test procedure is quite similar to the test procedure shown in Fig. 7 (preshear and shear), but less time-consuming because a complete yield locus can be measured with one specimen.

APPLICATION

One common application of shear tests is flowability testing, e.g., for powder development or quality control. In the following examples are presented for both applications, i.e., product flowability testing and equipment design.

Modification of flow properties

Flow agents are applied to improve the flow behaviour of powders. An example is shown in Fig. 10, where the flowability, ff_c, of a crystalline bulk solid is plotted versus flow agent concentration. The product had to be improved by the addition of a flow agent because it exhibited a strong time consolidation effect. To find the optimal concentration, samples with different percentages of flow agent were prepared. With each sample a yield locus (instantaneous properties) as well as a time yield locus for a storage time of 22 hours (t = 22 h) has been measured with a shear tester. As can be seen from Fig. 10, the sample with the concentration 0.55 % resulted in the largest flowability both for instantaneous conditions and after time consolidation. An increase of flow agent concentration beyond 0.55 % reduced flowability.

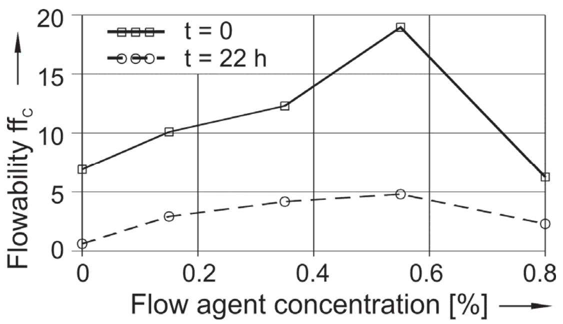

Fig. 10. Flowability, ff_c, as a function of flow agent concentration[1,9].

Equipment design

When a powder discharges from a hopper or silo, one must distinguish between mass flow and funnel flow (Fig. 11)[1,3]. In a mass flow silo every particle of the powder is moving whenever the outlet is opened. Mass flow is only possible if the hopper walls are steep and/or low enough in friction. If the latter is not the case, funnel flow will occur. In case of funnel flow, at first only the powder in a channel above the opening flows downwards. The powder located in the stagnant zones can be discharged only if the silo is completely emptied.

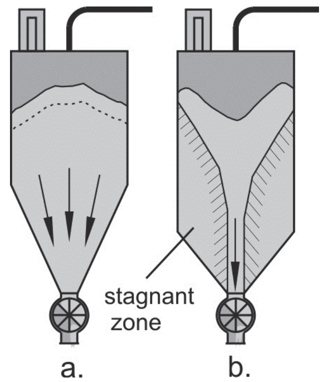

Fig. 11. a. Mass flow; b. funnel flow[1].

Equipment for powder handling, e.g., hoppers or bins, which is not designed adequately, may cause problems. Most frequent problems emerging during handling or storage of powders and bulk solids are[13]:

- Arching (Fig. 12.a). If the outlet is too small, a stable arch can form above the outlet and the flow stops.
- Ratholing, or piping (Fig. 12.b). In case of funnel flow, the powder in the stagnant zones can consolidate with time to such an extent that it will not be able to flow out after the flow zone has emptied out. The latter results in a "rathole" reaching from the outlet opening to the top of the filling.
- Segregation (Fig. 12.c): When filling a silo, the product can segregate across the cross-section of the silo, e.g., by the coarser particles collecting in the silo periphery. If funnel flow takes place at discharge, at first the material from the

centre (here: the fines) flows out, followed by the coarser material from the silo periphery

In a funnel flow silo all problems mentioned above can appear, while in the case of mass flow only the problem of arching must be considered[1,3]. For the design of a mass flow silo the slope of the hopper walls to ensure mass flow and the minimum outlet dimension required to avoid stable arches have to be determined. For this the flow properties of the powder have to be measured with an appropriate shear tester. The major quantity determining the required outlet dimensions is the unconfined yield strength. The hopper slope to achieve mass flow depends largely on the wall friction angle. The wall friction angle describes the friction between powder and hopper wall and can be measured with a shear tester applying a somewhat simpler procedure compared to a yield locus test[1,3].

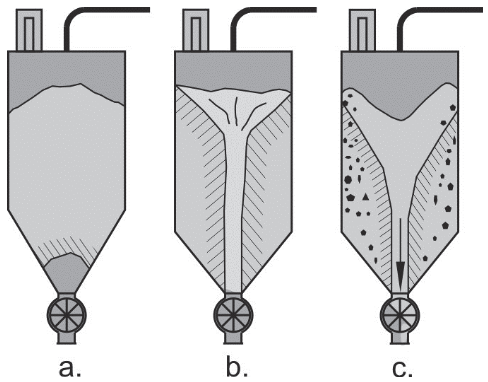

Fig. 12. Flow problems; a. arching; b. ratholing; c. segregation[1].

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