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A new Uniaxial Compression Tester: Development and Application

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Powder flow characterization with some of the conventional shear testers can be costly, time consuming and requires a trained operator, therefore the application of a cheaper, simpler, and sometimes faster uniaxial compaction tester (UCT) have often been suggested as an alternative. However, it has been known for many years that the results derived from the two methods are not necessarily the same, due to the lower state of compaction attained in the traditional design of UCT. Additionally, the traditional design of UCT tends to give a wide scatter in the results. To overcome these limitations of the UCT, this work developed an easy-to-use uniaxial tester in order to reduce the difference between flow properties reported by the two techniques. In this regard, flow functions of four powders in the cohesive and very cohesive range were measured with shear testers, conventional uniaxial compaction tester (UCT) and the new uniaxial tester at the University of Greenwich ("Greenwich Uniaxial Tester" or GUT). A method in the style of Janssen approach for correcting wall friction effect on the compaction stress of UCT was applied. Results showed the unconfined yield strength attained from GUT is in line with the results obtained from shear testers while the results from UCT are well below the shear tester results suggesting the advantage of the new GUT.

1. Introduction

It is estimated that 60% of products in chemical industry are manufactured as particulate solids and further 20% use powders as ingredients (Tomasetta 2013). All these materials have to be transported, conveyed, or handled. Therefore, the characterization of the flow behavior of powders plays an important role in industrial applications. The flow behavior of a bulk solid is dependent on the particle-particle interaction forces, however, it is not sufficient to quantify the particle-particle interaction alone because particle size distribution, shape, and surface properties of particles also each play a part, and their influences are difficult to correctly evaluate (Hamid Salehi, Lotrecchiano, et al. 2017; Salehi Kahrizsangi et al. 2015). A more practical way to approach the problem of describing the flow behavior of bulk solid is to consider it as a continuum medium. In this approach instead of forces between individual particles, stresses are regarded on boundary areas of volume elements. In continuum mechanics approach, the stress distribution inside a bulk solid is described by using the Mohr–Coulomb analysis, according to which the local state of stresses is represented by Mohr's circle on normal stress, σ, and shear stress, τ, plane. The intersection of the Mohr's circle with the normal stress, σ, axis determines the failure normal stress, σ_c, as well as major consolidation stress, σ_1 (Salehi, Poletto, et al. 2019). The flow properties of powder are usually reported by using the ratio between σ_c and σ_1, which is called the flow function (Schulze 2008).

The most trusted means of producing this flow function is to use a direct shear tester, there are several such testers available in market to measure flow function of bulk materials, e.g. Jenike shear tester (Schulze 2008), Schulze shear tester (Schulze 2008), Brookfield Powder Flow Tester (PFT) (Berry et al. 2014) and Anton Paar Powder tester (Hamid Salehi, Barletta, et al. 2017; Salehi et al. 2018). The way in which these testers work, requires several shear tests to be performed to obtain each point on the flow function – typically a minimum of 9 shear tests but more often 15 to 30, which requires a good deal of skilled labor for manual testers (i.e. Jenike shear tester) or a sophisticated machine in the case of automatic testers (i.e. PFT). These direct shear testers also require machinery that produces two orthogonal directions of movement, usually vertical translation and either horizontal translation or rotational about a vertical axis. As an alternative, the uniaxial compression tester represents a simpler method to obtain flow functions, in principle requiring only one direction of movement and producing a point on the Flow Function from each single test. However, the flow functions attained from a uniaxial tester usually exhibited lower flowability classifications when comparing to the results obtained from shear testers, since wall friction supports to some extend the external vertical consolidation stress applied on the sample (Guo et al. 2015; Luca Parrella et al. 2008). In order to overcome the wall friction effect Williams et al. (Williams et al. 1971) prepared powder samples by subsequent compaction of an increasing number of thin powder layers. The unconfined yield strength was derived from the experimental value of powder yield strength which extrapolated to an infinite number of layers. In another study, Maltby and Enstad (Maltby & Enstad 1993) wrapped the powder sample in a flexible membrane and added oil between the mould wall and membrane to decrease wall friction. Both studies reported the flow function values obtained from their methods close to one obtained from the Jenike shear tester. The authors concluded that problem is associated with the mould wall friction, often referred to as the Janssen effect (Nedderman 1992) causing non uniformity of the major consolidation stress, σ_1, which reduces exponentially with respect to the specimen depth. Also, it is common to find that the uniaxial tester produces a high degree of scatter in the data (Fitzpatrick & Descamps 2013).

Another limitation with using a direct shear tester is in regard to studies of powder caking. To clarify what this is, caking means large increases in strength in a bulk solid normally caused by development of a bonding mechanism between the particles, typically due to chemical reaction, moisture interaction, plastic flow or some other mechanism that causes permanent change to the particles. Such caking leads to very great increases in unconfined failure strength, often from one to three orders of magnitude or even more. This is very much greater than the “time consolidation” strength increase that occurs with most cohesive powders when left under consolidation stress for a period of time, due to rearrangement and settlement of particles that will often give a 10% to 50% increase in unconfined failure strength. Direct shear testers require a sensitive measuring system to resolve shear strength measurements to the accuracy needed for determining Flow Function, which is incompatible with the much larger measurement range that is required for measuring the strength of caked materials. Additionally, caking often requires some weeks of time to occur, so making caking measurements at several different stresses, under varying conditions of (for example) humidity and temperature for varying periods of time, to explore the conditions required to avoid caking, would require a single direct shear tester to be tied up for many months, or the use of multiples of these relatively expensive machines. For this reason, it is not economical to use such direct shear testers for caking measurements.

The practice that has evolved at The Wolfson Centre for caking studies, is therefore to use a traditional cylindrical uniaxial test. Because the test cell is very simple, many individual cells can be loaded, different stresses applied and then left in a single climate chamber for varying periods of time, in this way, many different test conditions can be created simultaneously, and the cells of caked sample then transferred to the uniaxial tester to make the cake strength measurement. However, the limitations of the effects of wall friction, and substantial scatter in the measurements, are still a problem with this test (Salehi Kahrizsangi et al. 2016).

Based on this problem, the purpose of this research work was to develop a simple, fast and easy to use new uniaxial tester, with minimised wall friction effects for better repeatability and accuracy; the result is the Greenwich Uniaxial Tester (GUT). The tester was successfully used for measuring caking strength of detergent powder in another study (Salehi, Berry, et al. 2019). In this study, the flow functions of 4 different powders (calcium carbonate, iron ore with 3% moisture content, titanium dioxide, and barite) were measured with GUT and UCT at the same level of consolidation stress (ranging from 10 to 130 kPa). Furthermore, the flow function curves from GUT and UCT were compared with the ones obtained from the Brookfield Powder Flow Tester (PFT) at low level of consolidation stress and with the Jenike type shear tester at the higher consolidation stresses. Although the main purpose of the tester is to facilitate caking tests, the testing of it for measurement of Flow Function under non-caking conditions was undertaken to ensure that it measures the same information as direct shear cells. In contrast to the conventional shear tester, the GUT has potential application as a very high stress flow function tester.

2. Material and methods

2.1 Greenwich Uniaxial Compaction Tester (GUT) - A novel and easy-to-use force displacement uniaxial tester for measuring quantitatively the failure strength of powder was developed with some similarity to the Johanson hang-up Indicizer (Bell et al. 1994) and the caking tester developed at the University College Cork (Fitzpatrick et al. 2010). It overcomes the wall friction problems of the conventional cylindrical uniaxial compaction tester (UCT) since it has a low height to diameter ratio to minimize the wall friction effect during consolidation.

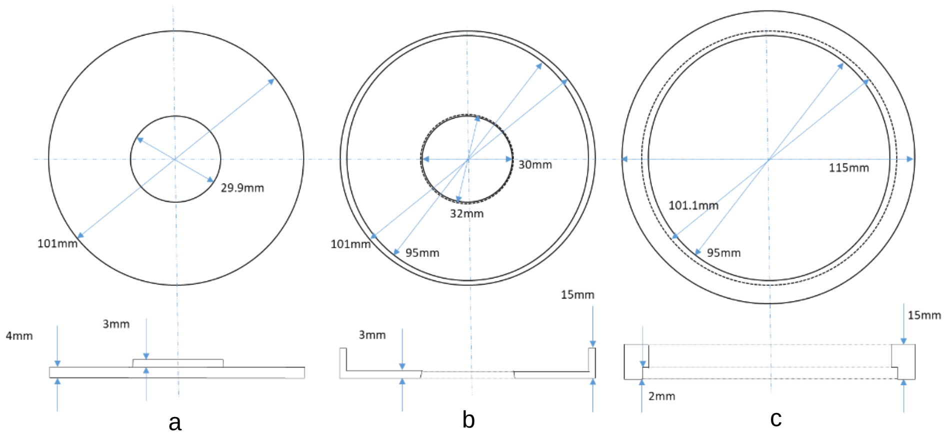

Figure 1. Greenwich Uniaxial Compaction Tester (GUT) dimension a) cell base, b) cell and c) *mould*

The developed tester has a defined location of the failure plane to maximise repeatability and it needs much smaller sample volume (around 75% reduction) compared to the UCT. The GUT consisted of a 95 mm internal diameter cell with a 30 mm diameter circular hole at its centre (Figure 1b). The hole is closed by 29.9mm protrusion in the cell base, in order to prevent powder falling out of the cell during consolidation (Figure 1a). A mould with the external diameter of 115 mm was designed for better filling the cell (Figure 1c). The mould was placed on the cell, and powder was poured into the tester and the excess amount was scraped off. Then the lid placed on the powder bed and loaded with different dead weights to achieve the desired consolidation stress (ranging from 10 to 130 kPa). After consolidation for a few seconds, the cell base was detached from beneath of the cell and then centred below a cylindrical punch (30 mm diameter) attached to a texture analyser to perform a failure strength measurement. Failure strength measurements were performed under quasi-static conditions and the texture analyser was programmed in a way that the punch was moved down at a constant speed of 0.4 mm/s and the force necessary for the punch to penetrate and push out the plug of the consolidated sample, through the hole in the cell base, was registered and reported. An example of stress-displacement curve during cake strength measurement is depicted in Figure 2. As can be seen, the peak is easily identified.

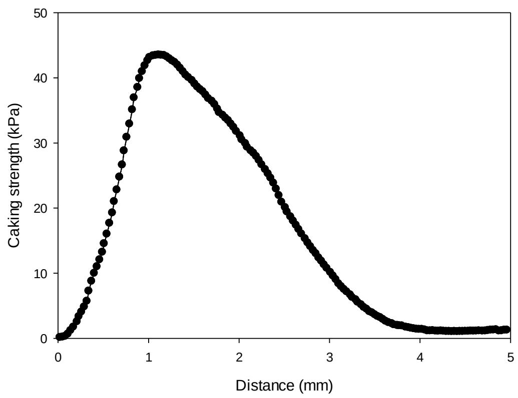

Figure 2. An example of stress-distance curve during cake strength measurement

2.2 Traditional Conventional (cylindrical) Uniaxial compaction test – in this technique the powder is poured into a cylindrical mould, in our experiments made of plastic, and then compacted under a defined normal stress, σ_1. After removal of the applied compacted normal stress, the cylindrical plastic mould is retracted in order to attain a consolidated free-standing powder sample with no lateral constraint. The consolidated powder is subjected to an increasing normal compressive force acting in the same direction of the compacted normal force, in order to attain the maximum force necessary to break the sample. The value of unconfined yield strength was determined by determining the maximum normal compressive force necessary to break the sample. The unconfined yield strength value, σ_c and major principal stress, σ_1 defines one point in the flow function graph in Figure 3. Repeating this procedure at different consolidation stress several times (at least 5 or

6 tests at each stress to get a reliable median, since the repeatability of this method is very low) enable us to draw a flow function curve.

2.3 Brookfield Powder Flow Tester (PFT) - The instantaneous flow function (the ratio between unconfined yield strength and major principal stress during consolidation) of the powders were also measured with the Brookfield Powder Flow Tester (PFT). The flow functions are important because their representation is the means by which powder flowability is usually reported and classified, according to the Jenike classification (Schulze 2008) or in other words according to the flow factor value, $ff = \sigma_1 / \sigma_c$. The classes generally considered are free-flowing (ff>10), easy-flowing (4<ff≤10), cohesive (2<ff≤4), very cohesive (1<ff≤2) and hardened or non-flowing materials (ff≤1). It is in the "hardened" category where most caked materials fall, whereas material that have not suffered some form of transition of their surface, chemistry or contacts cannot exceed the very cohesive range. Figure 3 reports the relevant flow regions and the flow factor lines at the boundaries. The wall friction angle, φ_w, was measured with the PFT on a wall sample of the same material which uniaxial tester mould is made of. The maximum possible pre-shear stress possible to apply in the PFT is around 13 kPa, therefore for higher consolidation stress (up to 130 kPa) a Jenike type shear tester was used. The detailed experimental procedure to measure material flow properties as well as powder wall friction with the PFT and Jenike shear tester are reported elsewhere (H. Salehi et al. 2017).

2.4. Wall friction correction factor for conventional (cylindrical) uniaxial compaction tester - In order to consider the effect of wall friction exerted from uniaxial tester mould on powder, a force balance on a differential slice element of the sample under consolidation (in the style of Janssen approach (Nedderman 1992) was applied. The effective normal consolidation stress, σ_{ef}, on the powder at the uniaxial cylinder could be calculated from:

$$\sigma_{ef} = \sigma_0 exp\left(-\frac{4\mu_W K_Z}{D}\right) + \frac{\rho_b\, gD}{4\mu_W K_Z}\left[1 - exp\left(-\frac{4\mu_W K_Z}{D}\right)\right] \qquad (1)$$

where σ_0 is the applied compaction stress on the sample, μ_w is the wall friction coefficient measured with the Powder Flow Tester (PFT) (Berry et al. 2014) and used to consider the effect of wall friction on the effective normal consolidation stress, K is the ratio between the radial and the vertical principal stresses which is not possible to directly measure at our facility, hence the estimation of K value considering application of the Mohr-Coulomb analysis could be attained from $k = \frac{1-sin\,\varphi_e}{1+sin\,\varphi_e}$, φ_e is the effective angle of internal friction measured with the PFT, z is the vertical coordinate, D is the mould diameter, ρ_b is the powder bulk density and g is the acceleration due to gravity. The second term in the equation is due to the sample weight which could be neglected due to the fact that the uniaxial test usually conducted at high level of consolidation stress. For correcting the unconfined yield strength value, the hypothesis is that the consolidated powder bed starts to break from the middle of powder bed. Hence the unconfined yield strength of materials was calculated considering half weights of powder bed. The modified value of unconfined yield strength and major compaction stress are reported in Figure 3.

3. Results and discussion

The flow functions of all the tested powder measured with UCT, GUT, PFT and Jenike type shear tester are reported in Figure 3. Each data point in Figure 3 is the average of three independent experimental results and the error bars representing the standard deviation between them. The flow functions for iron ore and barite (Figure 3a and 3b) obtained with the PFT lay within the cohesive region. The flow function of the powders tended to fall into the classification of easy-flowing and free flowing at the higher consolidation stress measured with the GUT, UCT and Jenike shear tester. The flow function of titanium dioxide (Figure 3c) obtained with all techniques crosses different regions of flow behaviour from very cohesive to easy flowing, showing an apparent better flowability as the major principal stress σ_1 increases. Calcium carbonate (Figure 3d) showed the better flowability compare to the other tested powders and classified as easy and free flowing on the range of tested consolidation stresses.

A general inspection of the figure reveals the good agreement between the flow functions of the powders measured with PFT, Jenike and GUT while the results from the UCT is well below the others for three out of the four powders. Furthermore, the standard deviation of the results obtained from GUT is smaller than those from conventional uniaxial tester (UCT). These experimental results suggest the soundness of the newly developed uniaxial tester, to be used as a more reliable method for measuring flow properties of particulate solids than the traditional cylindrical uniaxial compaction tester, under circumstances where the use of direct shear testers such as Jenike or PFT is impractical for reasons of capital cost or equipment tie-up for long time consolidation or caking studies.

In order to assess whether the differences between the flow functions indicated by the UCT as opposed to the other methods (PFT, Jenike and GUT) could be explained by wall friction giving different effective powder compaction values in the two experiments, results from the uniaxial compaction tester were recalculated by considering the effect of wall friction on the stress acting during compaction step. The UCT calculated flow function according to Eq (1) are depicted as solid filled squares in Figure 3. The recalculated UCT flow function is close to the PFT and Jenike flow functions for only the easy flowing powder (calcium carbonate, for which the initial deviation was already much smaller), suggesting the utility of the wall friction correction factor approach is not universal. For the other three powders, applying the correction does reduce the deviation, but does not produce anything approaching agreement with the three other measuring methods. The applied correction procedure requires the knowledge (or assumption) of the *K* value which needs the value of effective angle of internal friction measured with a shear tester, furthermore the approach need the knowledge of angle of wall friction measurable with shear cells. So, in practical terms, the wall friction correction procedure revokes the intrinsic simplicity of the uniaxial compression tests and is usually not pursued in industrial practice.

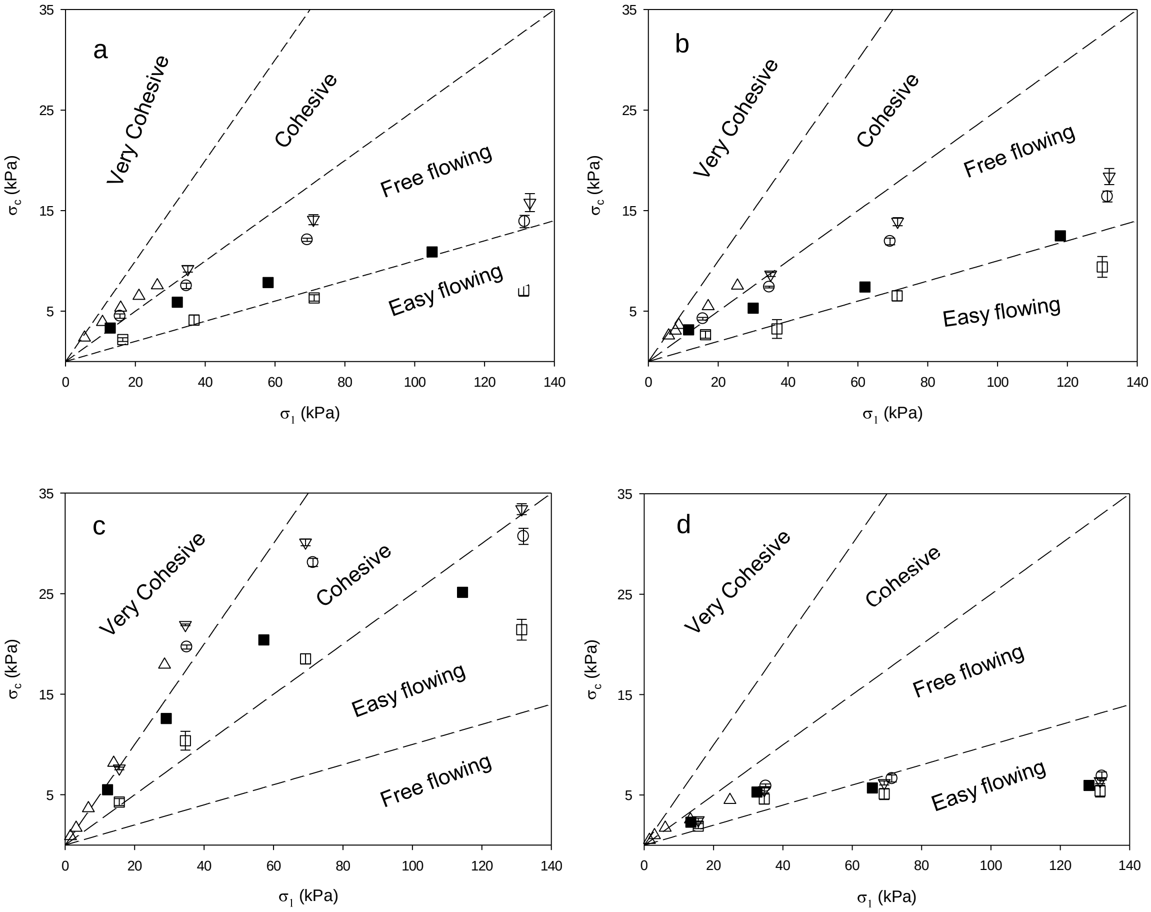

Figure 3. a) Iron ore, b) Barite, c) Titanium Dioxide and d) Calcium Carbonate ○ GUT, ▽ Jenike shear tester, △ PFT, □ UCT, ■ corrected UCT from equation 1.

Looking more closely at the performance of the UCT, the initial deviation of the UCT flow function was much greater for the other three tested powders. Even the corrected UCT flow functions, (cohesive and very cohesive), were well below the PFT and Jenike flow functions results except at the lowest tested consolidation stress. A possible reason for this observation is given by Parrella et al (L. Parrella et al. 2008). They attributed the different results between the corrected UCT and Jenike to the different stress histories in the sample preparation during the compaction steps for the two testers. However, it is not clear why this may apply at lower stresses but not higher ones.

Another phenomenon that might justify the difference between the unconfined failure strength of conventional uniaxial tester and shear tester results is anisotropy (Li & Puri 1996; H. Salehi et al. 2017). In conventional uniaxial compression tester, the materials retain their compression history and can provide lower shear stresses when the shear direction is different during the computation stress and the failure phase. A possible consequence is the apparent lower material shear failure measured with the conventional uniaxial tester than that with the Brookfield Powder Flow Tester.

4. Conclusion

A new and easy-to-use uniaxial tester was developed at the University of Greenwich and a comparison was performed between flow functions attained from this tester with PFT, Jenike and conventional cylindrical uniaxial compaction tester for four powders. The GUT gave unconfined yield strengths in good agreement with the Jenike and PFT shear testers, while the results from UTC was much lower. Recalculation of stress state of the sample in the uniaxial compression tester, taking into account the mould wall friction and the sample half weight, gave a flow function close to Jenike and PFT only for the free-flowing powder and not for the other three powders. The difference between the tested apparatuses could be attributed to the higher wall friction and anisotropy effect in the conventional traditional uniaxial tester.

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References

Bell, T.A. et al., 1994. Practical evaluation of the Johanson Hang-Up Indicizer. *Bulk Solids Handling*, 14(1), pp.117–125.

Berry, R., Bradley, M. & McGregor, R., 2014. Brookfield powder flow tester - Results of round robin tests with CRM-116 limestone powder. *Proceedings of the Institution of Mechanical Engineers, Part E: Journal of Process Mechanical Engineering*, 229(3), pp.215–230.

Fitzpatrick, J.J. et al., 2010. Comparing the caking behaviours of skim milk powder, amorphous maltodextrin and crystalline common salt. *Powder Technology*.

Fitzpatrick, J.J. & Descamps, N., 2013. Comparison of a Uniaxial Force Displacement Tester with a Ring Shear Tester for Assessing Powder Cake Strength. *Particulate Science and Technology*, 31(2), pp.199–203.

Guo, Z. et al., 2015. Study of flow characteristics of biomass and biomass-coal blends. *FUEL*, 141, pp.207–213.

Li, F. & Puri, V.M., 1996. Measurement of anisotropic behavior of dry cohesive and cohesionless powders using a cubical triaxial tester. *Powder Technology*, 89(3), pp.197–207.

Maltby, L. & Enstad, G., 1993. Uniaxial tester for quality control and flow property characterization of powders. *Bulk Solids Handling*, (1), p.135/139.

Nedderman, R.M., 1992. *Statics and Kinematics of Granular Materials*, Cambridge University Press.

Parrella, L. et al., 2008. Comparison between a uniaxial compaction tester and a shear tester for the characterization of powder flowability. *KONA Powder and Particle Journal*.

Parrella, L. et al., 2008. Comparison between a Uniaxial Compaction Tester and a Shear Tester for the Characterization of Powder Flowability. *KONA Powder and particle*.

Salehi, H., Berry, R., et al., 2019. Development and application of a novel cake strength tester. *Powder Technology*, 350, pp.36–42.

Salehi, H., Lotrecchiano, N., et al., 2017. Dust Release from Aggregative Cohesive Powders Subjected to Vibration. *Industrial & Engineering Chemistry Research*, 56(43), pp.12326–12336.

Salehi, H. et al., 2018. Experiments and simulation of torque in Anton Paar powder cell. *Particulate Science and Technology*, 36(4).

Salehi, H., Barletta, D., et al., 2017. On the use of a powder rheometer to characterize the powder flowability at low consolidation with torque resistances. *AIChE Journal*, 63(11), pp.4788–4798.

Salehi, H., Poletto, M., et al., 2019. Predicting the silo discharge behavior of wood chips - A choice of method. *Biomass and Bioenergy*, 120, pp.211–218.

Salehi, H., Barletta, D. & Poletto, M., 2017. A comparison between powder flow property testers. *Particuology*, 32.

Salehi Kahrizsangi, H. et al., 2015. Dust generation in vibrated cohesive powders. *Chemical Engineering Transactions*, 43.

Salehi Kahrizsangi, H., Barletta, D. & Poletto, M., 2016. Mechanical properties of agglomerates produced by the mechanical vibration of cohesive powders. *KONA Powder and Particle Journal*, 2016(33).

Schulze, D., 2008. *Powders and bulk solids : behavior, characterization, storage and flow*, Springer.

Tomasetta, I., 2013. *The effect of temperature on flow properties of powders*. PhD thesis, University of Salerno.

Williams, J.C., Birks, A.H. & Bhattacharya, D., 1971. The direct measurement of the failure function of a cohesive powder. *Powder Technology*.