AL16 - A Review of Powder Characterization

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Abstract

When a process modification involves a new, or a partly unknown material, it is very important to establish a good basis for the material characteristics. If a material sample is available it is possible to measure various material parameters and characterize the material perfectly. An accurate measurement of only a few material parameters will allow for a better selection of the necessary process steps for the material handling and selection of the right storage equipment. In this paper different ways to characterize powder and bulk material are introduced. This system is applied to a sample material, and to fractions of this material. Fractions of a material will behave differently compared to the complete material sample. The importance of a homogenous material for the design of the storage and handling system is discussed based on a simple schematic.

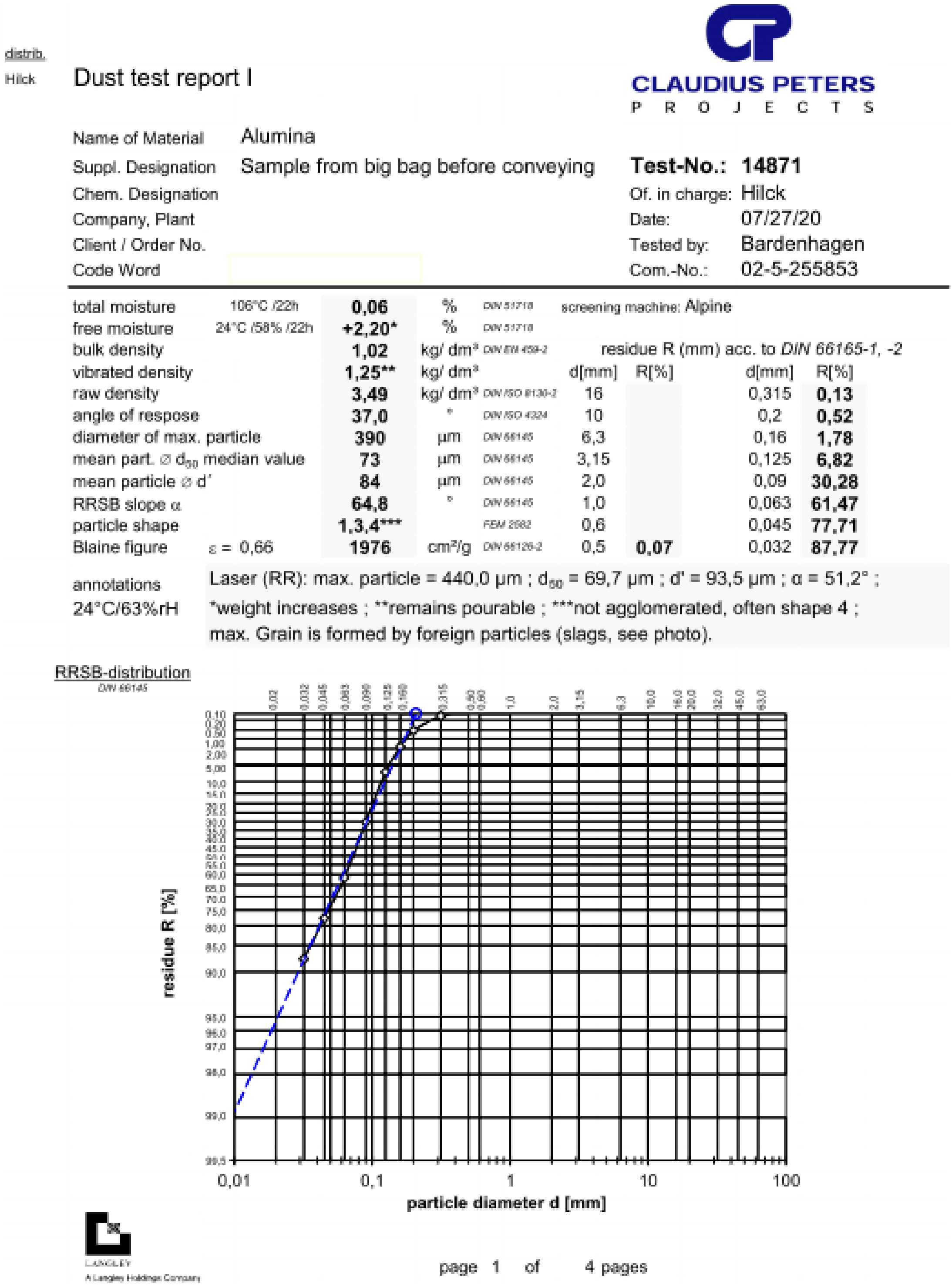
Keywords: Alumina handling, storage, material transport.

1. Introduction

A specialist company is requested by a plant operator or his planner to submit an offer for the construction of a sub-plant within a larger plant, e.g. a cement or aluminium producing plant. In addition to WHAT and FOR WHAT, the specifications attached to the request contains all basic requirements of the planned plant section. In the case of the bulk material processing plants considered here, this also includes the specification of basic bulk material parameters. However, these are generally limited to information on the average particle diameter, if necessary also the particle size distribution, the solid and bulk density as well as information on the chemical composition of the product entering the sub-plant. However, the bulk material behaviour changes along a process depending on the local handling/stressing situation, i.e. depending on the external forces acting on the bulk material in relation to the inter-particular forces, sometimes significantly. Furthermore, abrasion, grain destruction and/or segregation, operating temperatures deviating from the ambient conditions as well as the type and composition of the (gaseous) ambient medium have an additional influence on the actual bulk material behaviour. For a serious and reliable system project planning, the bulk material data listed in the specifications is therefore generally not sufficient. The specialist company inquired will therefore request a "representative" bulk material sample, quantity: $\gtrsim 10$ dm^3, from the plant operator for their own laboratory tests and measure the product characteristics that are relevant to the task from their point of view.

2. Measuring of Bulk Material Properties

Figures 1 and 2 show the basic version of a dust test report, as used in a similar way by various specialist companies, for a sandy alumina.



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Dust test report I

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Name of Material	Alumina
Suppl. Designation	Sample from big bag before conveying
Chem. Designation	
Company, Plant	
Client / Order No.	
Code Word	

Test-No.:	**14871**
Of. in charge:	Hilck
Date:	07/27/20
Tested by:	Bardenhagen
Com.-No.:	02-5-255853

total moisture	106°C /22h	**0,06**	%	DIN 51718	screening machine: Alpine
free moisture	24°C /58% /22h	**+2,20***	%	DIN 51718	
bulk density		**1,02**	kg/ dm³	DIN EN 459-2	
vibrated density		**1,25****	kg/ dm³		
raw density		**3,49**	kg/ dm³	DIN ISO 8130-2	
angle of respose		**37,0**	°	DIN ISO 4324	
diameter of max. particle		**390**	µm	DIN 66145	
mean part. ⌀ d_{50} median value		**73**	µm	DIN 66145	
mean particle ⌀ d'		**84**	µm	DIN 66145	
RRSB slope α		**64,8**	°	DIN 66145	
particle shape		**1,3,4*****		FEM 2582	
Blaine figure	ε = 0,66	**1976**	cm²/g	DIN 66126-2	

residue R (mm) acc. to *DIN 66165-1, -2*

d[mm]	R[%]	d[mm]	R[%]
16		0,315	**0,13**
10		0,2	**0,52**
6,3		0,16	**1,78**
3,15		0,125	**6,82**
2,0		0,09	**30,28**
1,0		0,063	**61,47**
0,6		0,045	**77,71**
0,5	**0,07**	0,032	**87,77**

annotations
24°C/63%rH

Laser (RR): max. particle = 440,0 µm ; d_{50} = 69,7 µm ; d' = 93,5 µm ; α = 51,2° ;
*weight increases ; **remains pourable ; ***not agglomerated, often shape 4 ;
max. Grain is formed by foreign particles (slags, see photo).

RRSB-distribution
DIN 66145

LANGLEY
A Langley Holdings Company

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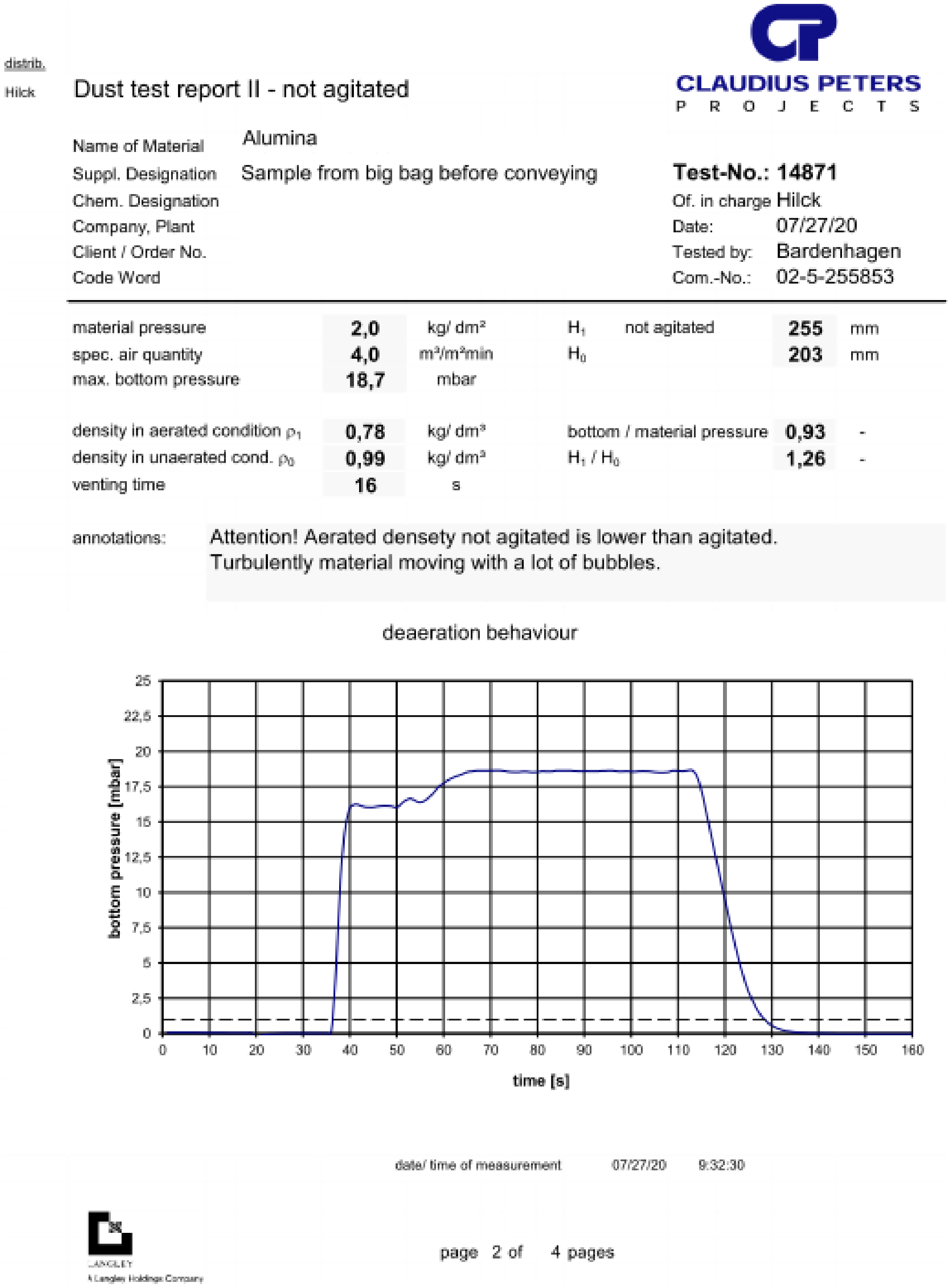
Figure 1. Dust Test Report, Page 1: Particle size and densities.



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Hilck

Dust test report II - not agitated

CLAUDIUS PETERS
PROJECTS

Name of Material	Alumina		
Suppl. Designation	Sample from big bag before conveying	**Test-No.:**	**14871**
Chem. Designation		Of. in charge	Hilck
Company, Plant		Date:	07/27/20
Client / Order No.		Tested by:	Bardenhagen
Code Word		Com.-No.:	02-5-255853

material pressure	**2,0**	kg/ dm²	H_1 not agitated	**255**	mm
spec. air quantity	**4,0**	m³/m²min	H_0	**203**	mm
max. bottom pressure	**18,7**	mbar			
density in aerated condition ρ_1	**0,78**	kg/ dm³	bottom / material pressure	**0,93**	-
density in unaerated cond. ρ_0	**0,99**	kg/ dm³	H_1 / H_0	**1,26**	-
venting time	**16**	s			

annotations: Attention! Aerated densety not agitated is lower than agitated.
Turbulently material moving with a lot of bubbles.

deaeration behaviour

date/ time of measurement 07/27/20 9:32:30

page 2 of 4 pages

Figure 2. Dust Test Report, Page 2: Fluidization and deaeration behavior of material.

Essentially, the granulometry of the bulk material, its different densities, e.g. loosely poured, aerated/fluidized bulk material density, the behaviour in the fluidized state under the two different stress states "unstirred", and "stirred", including the gas holding capacity when the gas supply is interrupted, and the flow behaviour on an aeroslide, are investigated. Details on the measuring methods can be found in the relevant standards and literature, e.g. [1-5]. Further investigations required depending on the task, e.g. on sorption, shearing, wear or grinding behaviour, are also carried out if required. A microscopic image of a particle fraction can among other things give an impression of the grain shapes occurring and the possible wear and tear of the plant. As already mentioned in section 1, the problem with using such a set of parameters is that the same bulk solid with the same parameters can behave very differently in different process steps or apparatus depending on the external stress. There is no direct assignment of certain behaviours to individual parameters, but several of these must be used, each with different weighting, to assess the current handling situation. This in turn means that the test report shown in Fig. 1 must always be interpreted in an application-specific manner. However, such an interpretation is greatly simplified if it is possible to compress the large number of product parameters influencing the bulk material behaviour into characteristic values, each of which summarises a part of these individual properties and allows bulk materials to be grouped into material classes with comparable behaviour under specific handling conditions.

3. Systems for Bulk Material Evaluation

In the following, some proven bulk material evaluation systems are presented and their application is discussed in the following section. The considerations are limited to dry bulk solids, i.e., products whose moisture content remains below a critical value. The influences of temperature, moisture, segregation and wear are discussed in [3-5].

3.1. Classification According to Geldart

Geldart [6] has developed a classification system based on its own and third-party measurements, which divides bulk solids into four groups - A, B, C, D - with regard to their fluidisation behaviour in gas/solid fluidised beds. Figure 3 shows this order scheme. The ordinate shows the difference between the particle density $\varrho_p \cong \varrho_S$ and the gas density ϱ_F, the abscissa shows a characteristic particle diameter d_S.

In the original work d_S is the diameter $d_{S.SD}$ of a sphere of equal volume and surface area. In the following, the mean particle diameter $d_{S.50}$ at a screen residue $R = 50\,\mathrm{M.\%}$ is used in a simplified way and with sufficient accuracy. For comparison purposes, quartz particles in different grinding fineness with $\varrho_p = \varrho_S = 2600\ \mathrm{kg/m^3}$ are considered here. Note: When flowing through a fluidised bed, the fluidising gas is divided in different ways into a bubble phase and a suspension phase, depending on the bulk material. The four bulk solids classes behave as follows [6, 7]:

Group A: Bulk materials consisting of fine-grained particles. The average grain size of the quartz reference materials is in the range: $d_{S,50} \cong (20 - 90)$ µm. Fluidized beds of group A materials expand homogeneously with increasing gas velocity to a range well above the minimum fluidization (→ gas velocity $v_{F.L}$ at the point of fluidization) before bubbling starts.
The bubble size remains relatively small. There is a maximum bubble size which, if exceeded, causes the bubbles to break up into smaller units. If the gas supply is abruptly switched off, the bed collapses very slowly at a speed which corresponds approximately to the gas empty tube speed in the suspension phase. All gas bubbles rise faster than the void gas in the suspension phase. The mean bubble size can be reduced in two ways: by a wide particle size distribution and/or a small mean particle size.

Group B: Bulk materials in the medium grain size range. The quartz reference materials are in the grain size range: $d_{S,50} \cong (90 - 650)$ µm. In contrast to group A, the formation of bubbles in group B bulk solids starts directly above the minimum fluidization. The bed expansion is low and if the gas supply is suddenly switched off, the bed collapses very quickly. The bubbles rise much faster than the void gas. There is no upper limit for bubble growth, it is generally limited by the dimensions of the apparatus (→ so called spouted bed).

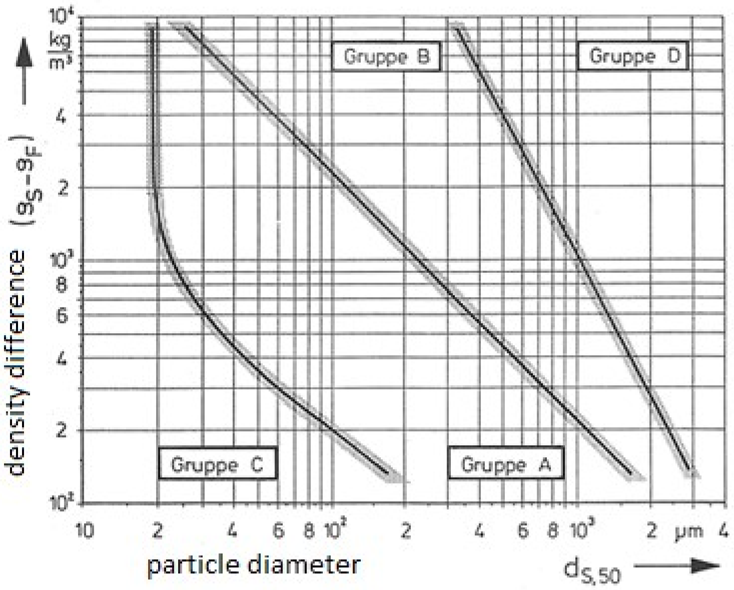

Figure 3. Solids classification according to Geldart [6].

Group C: This group includes bulk materials that are cohesive in some way. Particle size range of quartz reference bulk solids: $d_{S,50} \lesssim 20$ µm. "Normal" fluidization of these materials is extremely difficult. In small, smooth tubes, the bulk material is lifted as a whole/as a plug or the gas only blows out individual channels reaching from the inflow bottom to the bed surface (→ "Rat holes"). The reason for this is that the adhesive forces acting between the particles are noticeably greater than the forces exerted on the particles by the gas. Only by using mechanical stirrers can a more or less good fluidisation be forced.

Group D: These are bulk materials consisting of large and/or very heavy particles. The sizes of quartz reference bulk solids are above $d_{S,50} \gtrsim 650$ µm. The gas velocity in the suspension phase is relatively high. Most gas bubbles rise at a lower speed than the void gas. This means that the suspension gas flows through the gas bubbles from bottom to top. This results in a gas exchange mechanism between the suspension and bubble phase which differs from that of the bulk materials of groups A and B. Group D materials can form so-called "spouted beds."

The transitions between the individual groups are floating. The Geldart diagram implicitly describes the influence of two summarized bulk material parameters: gas-holding capacity and gas permeability of a particle composite. The gas holding capacity can be characterized by the venting time $\Delta\tau_E$ of a fluidized bed abruptly separated from the gas supply in a standardized apparatus under standardized conditions.

The influence of the width of the particle size distribution of a bulk solid on its fluidisation behaviour is only insufficiently covered by the Geldart diagram. An empirical adaptation/correction is described in [3, 4]. In general, as the particle size distribution widens, i.e. the proportion of fines increases, the gas holding capacity of a bulk solid increases, while the gas permeability decreases.

The classification according to Geldart allows a reliable assessment of the behaviour of the bulk material, especially if it is in a loosened state. This is the case in fluidised beds, in pneumatic conveying, especially in dense flow, in fast flowing bulk materials, e.g. on chutes, aeroslides, behind belt conveyors, when filling containers or when bridges, shafts and shafts in silos collapse, etc.

3.2. Flow Function According to Jenike

Jenike [8] has introduced the flow function FF_C for the qualitative description of the flowability and storage behavior of bulk solids. This is defined as the ratio of the compaction stress acting on the bulk material σ_1 to the uniaxial compressive strength, f_C generated by it., i.e. as

$$FFc = \frac{\sigma_1}{f_c} \qquad (1)$$

The idealized experiment in Figure 4 illustrates the importance of the characteristic FF_C. High FF_C-values describe a good, small values a bad flowability.

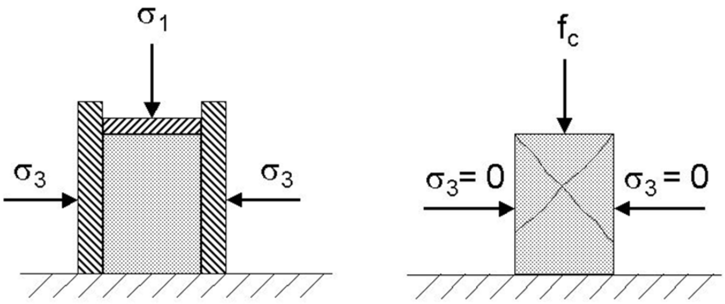

Figure 4. Idealised uniaxial test.

The main problem with the FF_C-Werts and the classification based on it is that extensive shear tests must be carried out to determine σ_1 and f_C. If the results of these experiments are available, they can also be applied directly to the respective problem. A classification of the bulk material for the evaluation of its expected behavior is thus no longer necessary. It makes more sense to be able to estimate the possible behavior of a bulk solid in a planned process even before the plant design. For this purpose, the FF_C-value can be determined from other, more easily accessible bulk material parameters. In [9, 10] FF_C is correlated with the parameters of an RRSB grain size distribution. The following applies:

$$FF_C = FF_c\left(n \cdot \sqrt{d_S^*}\right) \qquad (2)$$

with:		
	n	Gradient of the RRSB line ($= tan(\alpha_{RRSB})$),
	d_S^*	Particle diameter at a residue of $R = 36.8 \text{ m.}-\%$,

The approach considers both a "medium" grain size ($\rightarrow d_S^*$) and the width of the grain size distribution ($\rightarrow n$). Both parameters have a significant influence on the behavior of the bulk material. The following allocation/classification is used:

$10 \leq FF_C < \infty$	→	$20 \leq \left(n \cdot \sqrt{d_S^*}\right) < \infty$	free flowing	
$4 \leq FF_C < 10$	→	$10 \leq \left(n \cdot \sqrt{d_S^*}\right) < 20$	easy flowing	
$2 \leq FF_C < 4$	→	$5 \leq \left(n \cdot \sqrt{d_S^*}\right) < 10$	cohesive	(3)

$1 \leq FF_C < 2 \rightarrow 2.5 \leq \left(n \cdot \sqrt{d_S^*}\right) < 5$ very cohesive

$FF_C < 1 \qquad \left(n \cdot \sqrt{d_S^*}\right) < 2.5$ non-flowing, solidifying

The particle diameter d_S^* in eq. (3) must be used in the unit micrometer $[\,\mu\text{m}]$. Further investigations show that the integration of further bulk material parameters, e.g. the current bulk material density or the number of contact points $k \cong 3.14/\varepsilon_{SS}$ a particle with the surrounding grains, does not lead to a significant improvement in the accuracy of Equation (3).

The flow function FF_C allows an estimation of the bulk solid behavior, especially if it is present as a compact bulk solid bond, i.e. the individual particles forming the bond are in permanent contact with their neighboring particles. Such conditions exist in silos, moving bed reactors, mechanical transport systems and at all storage locations.

3.3. Compressibility According to Carr

To describe the compressibility of a bulk material the definition according to Carr [11] is used:

$$R_C = \frac{\varrho_{SR} - \varrho_{SS}}{\varrho_{SR}} \tag{4}$$

with: ϱ_{SR} - Vibration density,
ϱ_{SS} - loose bulk density, → see characteristic values in Fig. 1

The following classification is used:

$R_C < 0.2$	Bulk material shows good flowability, is free flowing Geldart group B-, D-, A/B-behavior	
$0.2 \leq R_C < 0.3$	Bulk material shows poor flowability, is cohesive Geldart group A-, C/A-behavior	(5)
$R_C \geq 0.3$	bulk material shows extremely poor flowability, is very cohesive, discharge/flow aids should be considered Geldart group C-, C/A-behavior.	

Bulk solids become more cohesive and therefore less free-flowing with increasing R_C-values. In particular, products with a high R_C tend to solidify considerably over time due to their loose structure and thus easy compactibility, e.g. due to applied loads, dead weight, etc., and require a higher energy input for restarting, which can result in start-up problems.

The compaction ratio R_C according to Carr allows, among other things, statements to be made on the storage and compacting behavior of a bulk material, on restarting a plant, e.g. a silo, after long shutdowns and to assess the expected time consolidation.

4. Example

4.1. Application on Whole Material Sample

Using the measured values taken from the test report in Figure 1a-e $\varrho_P \cong \varrho_S = 3490\ \text{kg/m}^3$, $\varrho_{SS} = 1020\ \text{kg/m}^3$, $\varrho_{SR} = 1250\ \text{kg/m}^3$, $d_{S,50} = 73\ \mu\text{m}$, $d_S^* = 84\ \mu\text{m}$ and $n = \tan(64.8°) = 2.13$ the bulk material examined there can be classified as follows:

- Geldart diagram: $\left(\varrho_S,\ d_{S,50}\right) = (3490\ \text{kg/m}^3, 73\ \mu\text{m})$ → The bulk material is a group A/B material and therefore easily fluidizable and ideally suited for aeroslide conveying.

The gas retention capacity/deaeration time is already relatively low. The alumina is located in the extended Geldart diagram between the two hatched areas, thus pneumatic dense phase conveying can only be realized by means of special bypass procedures or special measures.

- Jenike flow function: $\left(n \cdot \sqrt{d_S^*}\right) = \left(2.13 \cdot \sqrt{84\ \mu\text{m}}\right) = 19.52 \rightarrow FF_C \cong 10 \rightarrow$ The bulk material lies at the boundary from easy flowing to free flowing. Flow problems in flow-through containers, reactors or other accumulating aggregates are not to be expected.

- Evaluation acc. to Carr: $R_C = (\varrho_{SR} - \varrho_{Ss})/\varrho_{SR} =$ $(1250 - 1020)\ \text{kg/m}^3/1250\ \text{kg/m}^3 = 0.184 \rightarrow R_C < 0.2 \rightarrow$ The bulk material shows good flowability and is free-flowing; material does not tend to solidify over time, i.e. no start-up problems after plant shutdowns.

- Summary: The bulk material is not cohesive, flows well and does not tend to solidify over time. This is also confirmed by the measured slope angle, $\alpha_{SS} = 37.0°$. The expected storage behaviour in silos, containers etc. is unproblematic. Aeroslide transports can be realized without problems. Due to the position in the extended Geldart diagram, pneumatic dense phase conveying systems must be designed as bypass conveying systems or as system with a permanent fluidization. (Compare [12 to 16])

The assessments are as far as possible consistent and correspond with the practical experience gained from completed installations for this product.

4.2. Application on Fine Fraction

It is well known that the fine fraction of a material behaves completely different compared to the complete sample. Using the values from the test protocol we can derive limiting values from where on the classification will change for the material

- Geldart diagram: $(\varrho_S = 3490\ \text{kg/m}^3)$. Looking at the Geldart diagram this classification would only change, if the mean diameter reaches values of 10 µm. But the aeroslide behaviour will change massively on this way. The air retention time will vary with decreasing mean particle diameter.

- Jenike flow function: If the particle diameter decreases to values of 20 µm with the same width of distribution, there will be a different classification of the material. A wider particle size distribution would change the classification as well to a more cohesive behaviour.

5. Conclusions

The assessments are as far as possible consistent and correspond with the practical experience gained from completed installations for this product. With some values that are easy to assess from a particle size distribution it is possible to classify a material in terms of storage and handling. Some difficulties to collect the data persist, for example to judge the data from different measurement procedures.

6. References

1. David McGlinchey, (Ed.), *Characterization of Bulk Solids*, Blackwell, Oxford (2005)
2. Jonathan Seville, Chan-Yu Wu, *Particle Technology and Engineering*, Butterworth-Heinemann, Oxford, 2016.
3. Peter Hilgraf, *Projektierung und Auslegung von Schüttgutanlagen - Darstellung anhand von Beispielen*, Book under preparation.
4. Peter Hilgraf, Bulk Materials: Properties and Handling, Part 1 and 2. *ZKG Int*. 59(9) 2006, 58-69 and 59(10), 2006, 73-82.
5. Peter Hilgraf, *Schüttgut-Untersuchungs- und –Beurteilungsmethoden*, Claudius Peters Projects GmbH-Entwicklungsbericht, Buxtehude, 2002.
6. Derek Geldart, Types of gas fluidization, *Powder Technol*. 7, 1973, 285-292.
7. Otto Molerus, *Fluid-Feststoff-Strömungen*, Springer, Berlin, 1982.
8. Andrew Jenike, Storage and Flow of Solids, *Bull. No. 123, Utah Eng. Exp. Station*, Univ. of Utah, Salt Lake City ,1964.
9. Detlef Höhne., U. Schünemann, Untersuchung zum Einfluss der Korngrößenverteilungsparameter auf die Fließfähigkeit feinkörniger Schüttgüter, *European Symposium „Particle Technology, Volume B,* 1986“, Amsterdam, 968-980.
10. Peter Hilgraf, Reinhard Kleibs, Schüttguteigenschaften und Lagerzeit als Einflussgrößen bei der Auswahl des Silierungskonzeptes von Flugaschen, *VGB Kraftwerkstechnik* 6 1990, 479-486.
11. Ralph Carr, Evaluating Flow Properties of Solids, *Chem. Eng*. 18 1965, 163-168.
12. Mikel Leturia, et al, Characterization of flow properties of cohesive powders: A comparative study of traditional and new testing methods, *Powder Technol*. 253 2014, 406-423.
13. Andrew Jenike, Gravity Flow of Bulk Solids, *Bull. No. 108, Utah Engng. Exp. Station*, Univ. of Utah, Salt Lake City (1961).
14. Peter Hilgraf, *Pneumatische Förderung - Grundlagen, Auslegung und Betrieb von Anlagen*, Springer Vieweg Verlag, Berlin (2019).
15. Peter Hilgraf, Pneumatische Förderung - ein Überblick, Teil 1 und 2. *ZKG Int*. 46(1), 25-29, 46(3) 1993, 141-148.
16. Mark G. Jones, David Mills, Product classification for pneumatic conveying, *Powder Handl. Process.* **2**(2) 1990, 117-122.