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BISE Test Matrix Description Report

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Summary

The BISE test bench used for particle resuspension experiments was presented in the previous report (deliverable 23.31). The present report presents the parameters identified as playing a significant role in particle resuspension. Given the large number of factors involved in particle resuspension by airflow, this report also proposed a method to determine the test matrix, based on the use of test plans.

Approval

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1. Introduction

A detailed description of the BISE test facility where the particle resuspension experiments are to be conducted can be found in the previous report (deliverable 23.31). The present report provides a description and assessment of the proposed experimental measurement methodology, based on the use of test plans.

The report begins by presenting the measurement principle adopted and the limitations associated with the test procedure and instrumentation.

Given the large number of parameters to be studied, a rigorous experimental methodology needs to be used, based on the test plans presented in the third chapter.

2. Measurement and assessment methodology

2.1. Measurement principle

The quantity of resuspended particulate matter is defined as the powder mass entrained from the test surface during the experiment. It is necessary to choose between the two measurement techniques that can be implemented on the BISE test bench for assessing this quantity.

The first technique consists of determining the variation of the powder mass deposited on the test surface between the start and end of the experiment, with the quantity of resuspended particulate matter Δm corresponding to:

$$\Delta m = m_i - m_f \quad (2.1)$$

where m_i is the initial mass deposited on the test surface (g),

m_f is the final mass collected on the test surface (g).

For the purpose of the experiment, the mass of powder deposited is weighed along with the holder. Therefore, m_i is defined as the initial powder mass m_0 plus the mass of the holder m_{SE}. Likewise, m_f is defined as the mass of powder remaining on the holder, plus the mass of the holder.
The second technique consists of determining the mass of powder transferred to the collection filter (positioned downstream from the test surface with respect to the air flow) and the mass deposited between the test surface and the filter during the course of the experiment, with the total resuspended particulate mass corresponding to the sum of the two measured quantities, i.e.

$$\Delta m = m_F + m_D \quad (2.2)$$

where m_F is the mass of powder transferred to the filter (g),

m_D is the mass deposited between the test surface and the filter (g).

The proposed analysis technique for determining the quantity of suspended particulate matter is a fine gravimetric method (Sartorius MC 210P scale with 10 µg precision) that can be used to measure the total mass of suspended particulate matter. This measurement can then be used to calculate the resuspended fraction, defined as follows:

$$K_R = \frac{\Delta m}{m_0} \quad (2.3)$$

2.2. Detection limits

Given the large expected variability of the phenomenon studied, it is important to determine the experimental limitations of the proposed methodology so as to assign a sufficiently high level of confidence to the results obtained.

A first series of experiments is conducted to determine the detection limits of the measurement methods used. These detection limits are determined based on weight reproducibility measurements, according to a standardized protocol defined in ISO/CD 15767 (1999). This international standard describes a methodology for determining detection limits applicable to weight measurements of particulate matter collected on sampling filters. The limit values obtained correspond to the variation in mass below which no significant loss or collection of particulate matter may be observed.

For the first method (variation of powder mass deposited on test surface), reproducibility measurements are obtained by taking six successive weight measurements of six different powder deposits in random order, i.e. a total of 36 weight measurements. The powder deposits are conditioned in a plexiglass box throughout the experiment. Therefore, the detection limit on the test surface (LD_{SE}) takes into account both the precision of the scale and the influence of environmental parameters (temperature, humidity, displacement of powder deposits, etc.). The following value is obtained:

$$LD_{SE} = 0.1 \text{ mg}$$

This value corresponds to a suspended fraction K_R of 2.10^{-5} for an initial powder mass of 5 g deposited on the test surface.

For the second method (collection of resuspended powder), the detection limit on the collection filter (LD_F) is determined by performing intra and inter-filter reproducibility measurements. The following value is obtained:

$$LD_F = 2.8 \text{ mg}$$

This is approximately 30 times greater than the LD_{SE} value obtained with the first method. Moreover, this LD_F value corresponds to a resuspended fraction K_R greater than 6.10^{-4} (for an initial powder mass of 5 g deposited on the test surface), i.e. the fraction transferred to the filter.
It should be noted that this high LD_F value is mainly due to the significant size of the filter (204×253 mm^2) and its sensitivity to environmental conditions, particularly humidity.

The comparison of the two detection limit values leads us to discard the second method in experiments aiming to determine the total mass of suspended particulate matter. The method based on twofold weighing of the test surface reduces the maximum K_R value by a factor of approximately 90, and its ease of implementation allows better control and long-term reproducibility.
By comparing these results with those obtained by Fromentin (1989) under similar conditions, it can be seen that the LD_{SE} value determined here is quite satisfactory. Indeed, the maximum K_R value obtained by Fromentin can be estimated at approximately 10^{-4} for an initial powder mass of between 3 and 30 g. The gain achieved therefore amounts to a factor of at least 50.

All the test results presented hereinafter are obtained using the method based on twofold weighing of the test surface, referred to as DPS. For particle resuspension values very close to the detection limit LD_{SE}, it is arbitrarily decided that if $K_R \leq \frac{LD_{SE}}{2}$, then an assigned value equal to $\frac{LD_{SE}}{2}$ is used. If $K_R > \frac{LD_{SE}}{2}$, then the K_R value obtained is used.

2.3. Estimation of measurement uncertainties

For each test result obtained, an associated measurement uncertainty is calculated. For reasons justified in chapter 4 (large variability range of resuspended fraction K_R), the measurement uncertainty is calculated based on the decimal logarithm of the resuspended fraction K_R.

- The suspended fraction K_R is experimentally determined using the following equation:

$$K_R = \frac{m_i - m_f}{m_i - m_{SE}}, \quad (2.4)$$

where m_{SE} is the mass of the test surface.

The variance of the calculated Log K_R value can therefore be estimated as follows:

$$\sigma^2(\text{Log } K_R) = \left(\frac{\partial \text{Log } K_R}{\partial m_i}\right)^2 \sigma^2(m_i) + \left(\frac{\partial \text{Log } K_R}{\partial m_f}\right)^2 \sigma^2(m_f) + \left(\frac{\partial \text{Log } K_R}{\partial m_{SE}}\right)^2 \sigma^2(m_{SE}). \quad (2.5)$$

The test surface mass (m_{SE}), the test surface mass plus the initial powder mass (m_i) and the test surface mass plus the final powder mass (m_f) are weighed three times for each experiment. The value of Log K_R is determined from the mean value of the three weighing results. Variances $\sigma^2(m_{SE})$, $\sigma^2(m_i)$ and $\sigma^2(m_f)$ correspond to the square of the standard deviation of the three weighing results, i.e.

$$\sigma^2(m_{SE}) = \frac{n\sum m_{SE}^2 - \left(\sum m_{SE}\right)^2}{n \quad (n-1)}, \quad (2.6)$$

$$\sigma^2(m_i) = \frac{n\sum m_i^2 - \left(\sum m_i\right)^2}{n \quad (n-1)}, \quad (2.7)$$

$$\sigma^2(m_f) = \frac{n\sum m_f^2 - \left(\sum m_f\right)^2}{n \quad (n-1)}, \quad (2.8)$$

where n is the number of successive weighings (in this case, n=3).

The calculated variance of Log K_R should provide a good assessment of measured values, since variance values $\sigma^2(m_{SE})$, $\sigma^2(m_i)$ and $\sigma^2(m_f)$ are determined experimentally, and not theoretically based on potential sources of uncertainty (position of scale, etc.). This calculated variance corresponds to the dispersion of measured values with respect to the average, all raised to the power 2. The standard deviation of Log K_R, σ (Log K_R) is therefore referred to as the standard measurement deviation.

- The standard deviation of the dispersion of Log K_R, σ' (Log K_R) values, referred to as the experimental standard deviation, is calculated with the following equation, using the n resuspended fraction values obtained experimentally under identical initial conditions:

$$\sigma'(\text{Log } K_R) = \sqrt{\frac{n \sum \text{Log } K_R^{\,2} - \left(\sum \text{Log } K_R\right)^2}{n \quad (n \quad - \quad 1)}}, \quad (2.9)$$

where $\nu' = n-1$ is the degree of freedom.

These σ' (Log K_R) values can then be used to calculate a merged standard deviation (experimental standard deviation of mean measurement dispersion) which takes into account the number of tests σ_{exp}, i.e.

$$\sigma_{exp} = \sqrt{\frac{\sum \nu' \sigma' (\text{Log } K_R)^2}{\sum \nu'}}, \quad (2.10)$$

where $\nu_{exp} = \sum \nu'$ is the merged degree of freedom.

3. Test matrix

Given the complexity and expected variability of the physical phenomenon studied, and the large number of parameters possibly involved, an experimental methodology based on the use of test plans has been adopted. The test plans used will not be reviewed exhaustively here. The information given here is intended to provide a basic understanding of the work completed.

3.1. Test plans

In the rest of this report, the terms "factor" and "parameter" will be used interchangeably to refer to the various variables potentially affecting particle resuspension.

Test plans are used to define an experimental research methodology for obtaining an optimal amount of reliable information with a minimum number of tests (Droesbeke *et al*., 1997; Queffelec, 1999).

Since the present study involves approximately twenty parameters potentially affecting particle reentrainment, the experiments conducted must be specifically targeted using a rational approach. Test plans provide a basis for such an approach by clearly identifying the problems targeted and defining optimal test strategies and schedules based on target goals, imposed constraints and available resources.
As compared to the conventional and intuitive approach consisting of varying one parameter at a time, the test plan-based approach has the advantage of reducing the number, duration and cost of tests while optimizing the quantity of information obtained. However, such an approach imposes an indispensable condition: the test plan parameters adopted must be able to vary independently, at the expense of reducing the scope of analysis of certain parameters.

Four successive steps are required to implement a test plan:

- The first step consists of defining the problem targeted based on consideration of technical objectives and physical constraints (i.e. identification of influential parameters, determination of experimental domain, selection of characteristic responses).

- The second step consists of postulating a mathematical model (linear model with or without interaction, quadratic model, etc.) that describes the experimental domain considered, and then specifying a test strategy that determines the choice of test matrices (several test matrices can be defined to allow for different possible approaches).

- The third step consists of conducting the experiments and analyzing the results obtained.

- The fourth step consists of calculating mathematical model coefficients and comparing statistically significant results with available knowledge regarding the physical phenomenon studied.

  It is recalled that the present study aims to assess the main parameters affecting particle reentrainment from a polydisperse powder deposit by turbulent horizontal airflow.

Therefore, prior to establishing an initial test plan, it is necessary to identify the most relevant parameters to be studied. Each parameter selected must then be examined individually to determine the relevant experimental domain within the scope of the test plan.

3.2. Choice of parameters studied

A bibliographic study on particle reentrainment has led to the identification of approximately twenty parameters potentially influencing the phenomenon (Gensdarmes, Roynette, 2011). Choices must

therefore be made as to which parameters should be studied. The following has been taken into consideration:

- importance assigned to these parameters in the various theoretical and experimental studies present in the scientific literature,
- experimental limitations of the BISE test bench (speed and acceleration ranges, non-controllable parameters such as the ambient temperature and relative humidity in the test facility, etc.),
- independent parameter variation requirement imposed by the use of test plans (e.g. impossibility of independently varying the shape, density and mass of the powder).

Eight controllable (i.e. variable) parameters have been identified and chosen for the study.

- Four powder-related parameters:
  - particle size distribution of the powder, characterized by the mean particle diameter d_{50},
  - tap density of the powder (ρ_{tas}),
  - geometrical dimensions of the powder pile,
  - moisture content of the powder.
- One parameter associated with the deposition test surface:
  - roughness of test surface (Rug_{SE}).
- Three airflow-related and ambient parameters:
  - near-wall friction velocity v_*,
  - initial acceleration α,
  - exposure time Δt.

Two parameters that cannot be controlled due primarily to the facility's location are also measured during the experiments: ambient air temperature and relative humidity of airflow in the test facility.

In the laboratory where the experiments are conducted, mean temperature is 24 ± 8°C and relative humidity is 44% ± 26% RH).

Considering the information available and the experimental limitations involved, aluminium oxide (Al_2O_3) powders have been selected for the study. These powders possess certain physico-chemical properties similar to those of carbon powders possibly encountered in HTRs (particle size distribution, morphology, apparent density, etc. – see section 3.3.10). They also have the advantage of being readily (and inexpensively) available on the market with different particle size distributions. Furthermore, Al_2O_3 powders can be obtained with different shapes (i.e. rounded grains or sharp-edged particles), thus allowing for the possibility of varying the particle morphology parameter in future studies. The alumina powders used in the present study are marketed by Durmax.
There are subsequent plans to use graphite powders similar to those produced in HTRs. These powders will be developed in work package WP2.3.2, in collaboration with the University of Dresden. Experimental test conditions will be defined based on the results obtained using the test matrix presented here.

3.3. Determination of experimental domains

As previously indicated, the use of test plans imposes the need to vary parameters independently of one another. Therefore, each of the eight parameters selected has been examined individually to determine the relevant experimental domain under BISE test conditions and in accordance with the test plans.

Powder-related parameters

Particle size distributions of Al_2O_3 powders

The Al_2O_3 powders used have the advantage of possessing the following properties in common:

- particle density equal to 3.9 g/cm^3,
- apparent density ranging from 1 to 1.5 g/cm^3 (see subsection 3.3.1.3),
- sharp-edged particle structure as shown in Figure 3.1 below (SEM images of SPM 91 powder particles, shown at 500x magnification on the left and 2000x magnification on the right).

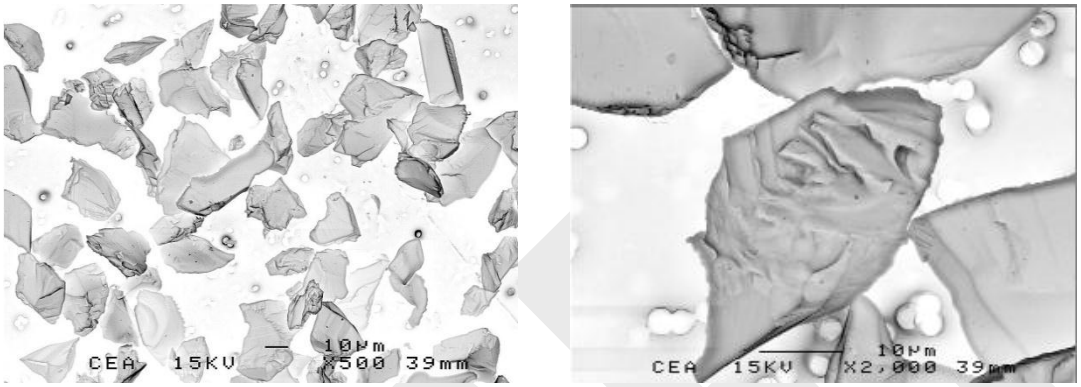

Figure 3.1 – Scanning electron microscope images of Durmax alumina powder particles (SPM 91)

Measurements taken with a Malvern Sysmex particle shape analyzer are used to determine the circularity of the particles (ratio between equivalent disc perimeter and particle perimeter). It is thus determined that SPM 91 alumina powder particles have a mean circularity index of 0.77, with a measurement dispersion of approximately 20 %. The powder therefore comprises of a low proportion of long-shaped particles.

Alumina powders are available with different particle size distributions, making it possible to scan particle sizes ranging from a few micrometers to approximately 100 µm. After selecting five alumina powders with polydisperse particle size distributions, Coulter Multisizer IIE measurements are used to verify that the target particle size range is effectively covered.

For this purpose, powder samples are suspended at low concentration in an electrolyte/glycerol solution. The solid particles to be analyzed are then passed through a calibrated orifice submerged in the electrolyte. As each particle enters the orifice, it displaces an electrolyte volume equal to its own volume, causing a change in base impedance proportional to the displaced volume. Therefore, each particle passing through the orifice generates an electric pulse with amplitude proportional to the particle's volume. The volume-equivalent particle diameter can thus be obtained. Consequently, the random passage of a large number of particles can be used to determine the particle size distribution of the entire sample.

Figure 3.2 shows the particle size distributions of the five alumina powders as a function of volume-equivalent diameter.

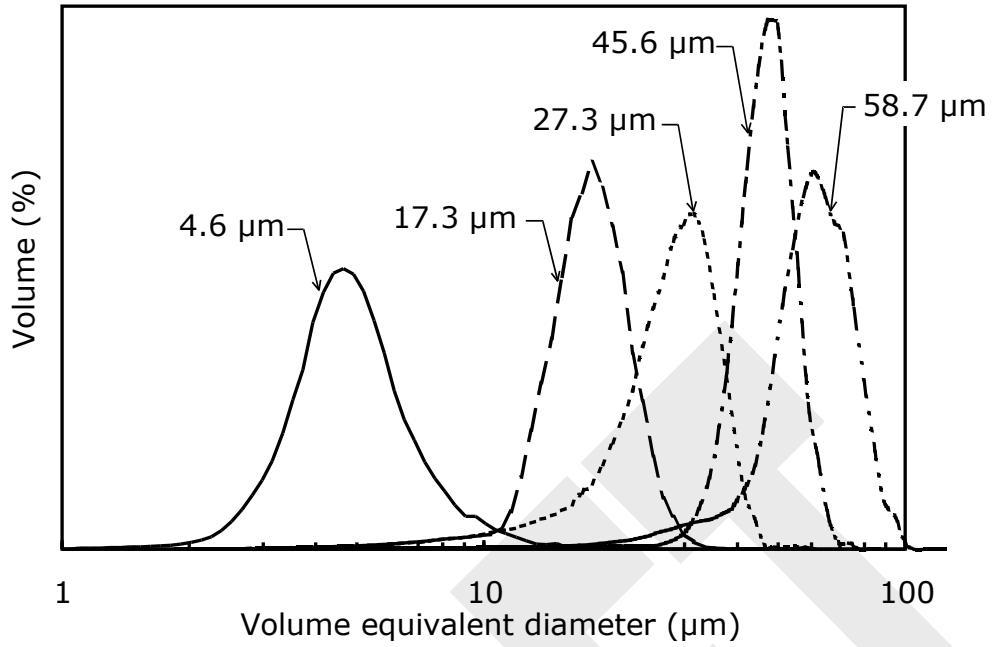

Figure 3.2 – Particle-size volume distributions of Durmax Al_2O_3 powders

As can be seen, the powders chosen effectively cover the particle size range from a few micrometers to 100 µm.

Furthermore, the standard geometric deviation values σ_g calculated from these particle size distributions (see Table 3.1) are practically constant regardless of the powder considered (mean value: 1.3 ± 0.1). The particle size distribution around the mean diameter d_{50} is therefore proportionally the same from one powder to another. Therefore, the powders chosen exhibit a homogeneous size distribution and low measurement dispersion.

Table 3.1 – Particle size distributions of Al_2O_3 powders

Durmax Al_2O_3 powder	*d_{50} (µm)*	*σ_g (-)*	*0% < (µm)*	*10% < (µm)*	*50% < (µm)*	*90% < (µm)*	*100% < (µm)*
SPM 102	**4.6**	**1.4**	1.0	3.2	4.6	7.2	22.0
SPM 91	**17.3**	**1.2**	2.0	12.8	17.3	22.5	58.0
SPM 84	**27.3**	**1.3**	2.0	16.0	27.3	35.6	54.9
SPM 52	**45.6**	**1.2**	2.8	36.4	45.6	54.8	73.4
SPM 47	**58.7**	**1.3**	4.0	40.6	58.7	75.5	99.6

In order for these particle size distributions to be properly represented, each powder must be homogenized in a Turbula T2C mixer for approximately 15 minutes before being sampled.

Moisture content of Al_2O_3 powders

The moisture content of the powders is determined using the conventional stove method. Three samples of each powder are weighed and placed in a stove at 180°C. The variation of the mass of the samples is then monitored over time. Before each weighing, the samples are removed from the oven and placed in a desiccator to allow the product to cool in the presence of dry air.

Figure 3.3 shows the variation with time of the evaporated water mass for alumina powders SPM 102, SPM 84 and SPM 47. The results are plotted with a confidence interval of 95%.

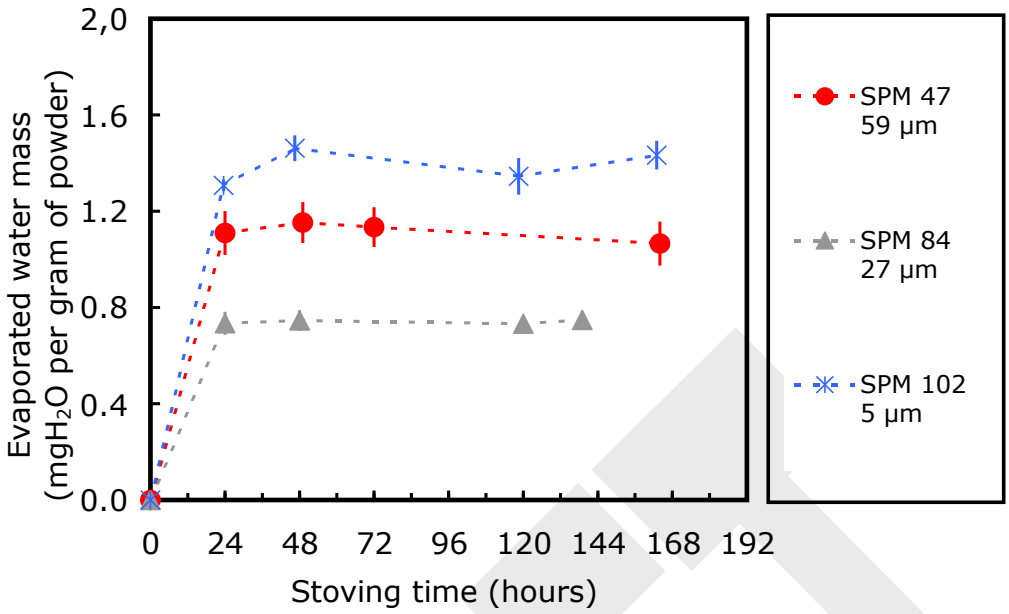

Figure 3.3 – Variation of evaporated water mass for different alumina powders (Stove at 180°C)

After 24 hours of drying, the evaporated water mass of each powder tends to stabilize and the variation coefficient becomes less than or equal to 3%. It can therefore be considered that after 24 hours, the powders contain no more water and are dry. Under these conditions, it is assumed that the moisture content is 0 mgH_2O per gram of powder.

Table 3.2 shows the moisture contents of the various Al_2O_3 powders under ambient conditions (i.e. T ~ 20°C, P = 1 bar, 40 < HR < 50%). This data is based on the results shown in Figure 3.3 at t = 24 hours, supplemented with additional measurements performed on the five powders after 24 hours of drying.

Table 3.2 – Moisture content of Al_2O_3 powders under ambient conditions

Durmax Al_2O_3 powder	H_20 content (mgH_2O/g)	Variation coefficient (%)	Number of samples
SPM 102 (d_{50}= 5 µm)	1.3	3	3
SPM 91 (d_{50}= 17 µm)	0.8	10	3
SPM 84 (d_{50}= 27 µm)	0.8	8	7
SPM 52 (d_{50}= 46 µm)	1.0	11	12
SPM 47(d_{50}= 59 µm)	1.1	7	10

The moisture content of powders SPM 102 and SPM 47 is approximately 1.2 mgH_2O/g, whereas that of powders SPM 91 and SPM 84 is slightly lower (0.8 mgH_2O/g). These slight differences should have no significant impact on the results.

The relevant experimental domain is therefore comprised between 0 and approximately 1 mgH_2O per gram of powder, with a variation coefficient of approximately 20% for the five powders. However, for technical reasons, experiments are only conducted at extreme values of this domain. Indeed, due to the difficulty of obtaining a powder with an intermediate moisture content (i.e. 0.5 mgH_2O/g), the trends observed in the statistical analysis of test results will be taken into account.

Considering that the dry powder samples must be placed inside the test facility during the experiments, the variation of the mass of these powders when placed in contact with ambient air must be studied so as to determine the water recovery rate. Powder samples previously dried in the stove

are therefore left in contact with the ambient air and subsequently weighed over a period of 24 hours after drying. The results are presented in Figure 3.4, in the same manner as in Figure 3.3.

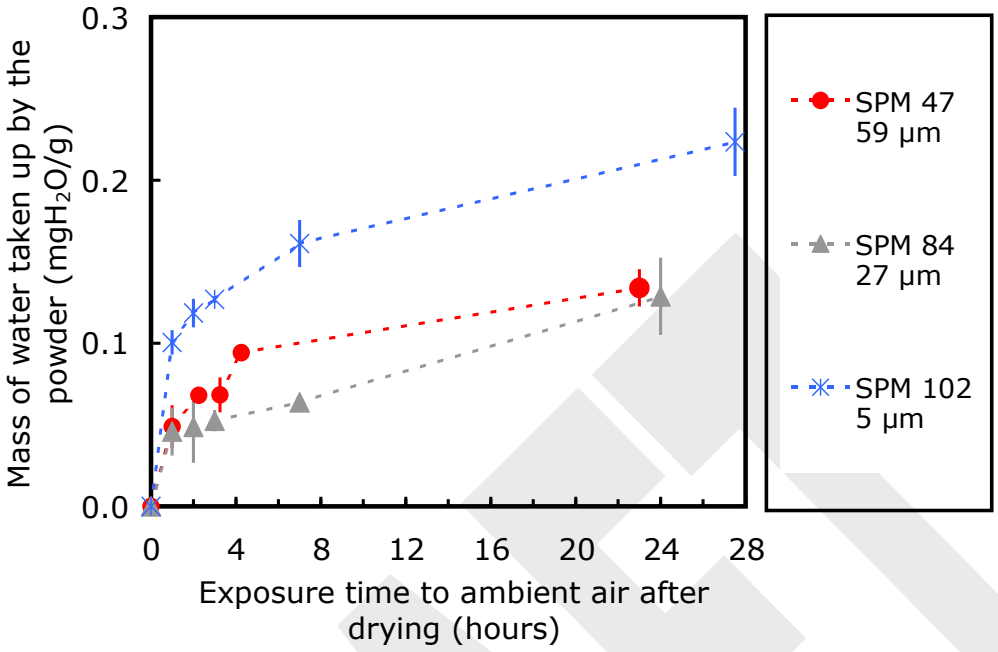

Figure 3.4 – Variation of water recovery rate of dry powders in contact with ambient air

Figure 3.4 shows the relatively slow water recovery rate of powders SPM 102, 84 and 47 in contact with ambient air. After 4 hours, the powders recover an average of 0.1 mgH_2O/g, i.e. 10% of their initial moisture content. After 24 hours, the percentage reached is only 15%. During the course of an experiment, the exposure time of the powder to ambient air is mainly determined by the exposure time of the powder deposit to the airflow. On average, Δt = 2 h corresponds to an exposure to ambient air for 3 hours (taking into account deposition and weighing times before and after the experiment). However, for K_R values of less than 10^{-2}, the recovered water mass must be taken into account since it is no longer negligible in comparison with the suspended particle mass. Based on the results shown in Figure 3.4, when performing an experiment on a dry powder sample, a moisture correction must be systematically applied to the final mass m_f collected on the test surface after the experiment (subtraction of recovered water mass).

Tap density of Al_2O_3 powders

Any volume containing powder particles is made up of solid matter and voids (i.e. pores and interstices). The volume in which these components are taken into account is referred to as the apparent volume (standard NF X 11-601, 1979). By reducing the quantity of voids (interstices) in the powder, it is also possible to decrease its volume. A simple method consists of compacting the powder to reduce its porosity.

The density of a bulk powder sample is referred to as the apparent density, and that of a compacted powder sample is referred to as the tap density.

The apparent density and tap density are determined using two methods:

- test tube method,
- powder tester method.

The first method consists of pouring the powder into a test tube via a funnel. The apparent volume (and apparent density) of each sample can then be determined. Compacted volumes are obtained by successively shaking each sample, causing the particles to rearrange into a smaller volume. Once the

volume has stabilized, it can be used to determine the compacted volume (and tap density) of the sample.
These measurements are then verified using a Hosokawa Micron powder tester, i.e. a device developed based on the work of Carr (1965), used to physically measure certain powder characteristics, including compressibility (ratio between apparent density and tap density). Contrary to the measurements obtained using the test tube method, these physical measurements (based on the same principles) have the advantage of eliminating operator uncertainties.

The table below lists the apparent and tap densities obtained with the test tube and powder tester methods for powders SPM 102, 91, 84, 52 and 47:

Table 3.3 – Apparent and tap densities determined with test tube and powder tester methods

Durmax Al_2O_3 powder	Apparent density (g/cm^3)		Tap density (g/cm^3)	
	Test tube (var. coeff. %)	Powder Tester (var. coeff. %)	Test tube (var. coeff. %)	Powder Tester (var. coeff. %)
SPM 102 (d_{50}= 5 µm)	1.0 (-)	0.9 (-)	1.8 (3)	1.7 (-)
SPM 91 (d_{50}= 17 µm)	1.2 (9)	1.2 (2)	2.0 (3)	1.8 (1)
SPM 84 (d_{50}= 27 µm)	1.5 (11)	1.4 (1)	2.0 (2)	2.0 (1)
SPM 52 (d_{50}= 46 µm)	1.6 (5)	1.5 (1)	2.1 (3)	1.9 (1)
SPM 47(d_{50}= 59 µm)	1.6 (4)	1.6 (1)	2.0 (-)	2.0 (2)

The two measurement methods yield very similar results. Nevertheless, the measurement reproducibility of the powder tester method (variation coefficient of 1 to 3 %) is better than that of the test tube method (variation coefficient of 2 to 11 %).
Regardless of the method used, the apparent densities of the five powders vary from 1.0 to 1.5 g/cm^3 (± 0.1) and the tap densities vary from 1.8 to 2.0 g/cm^3 (± 0.1). Since test plan requirements impose the use of overlapping domains, the experimental domain adopted is very narrow, i.e. 1.5 to 1.8 g/cm^3 (± 0.1).

Geometrical dimensions of powder pile

The present study must consider the shape of the powder pile formed by natural deposition. For this purpose, tests are performed in which powder is poured naturally through a funnel (Figure 3.5).

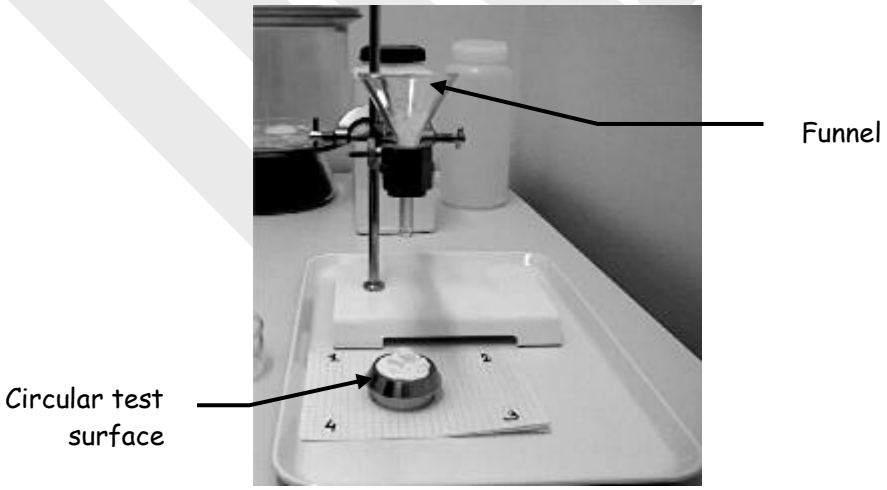

Figure 3.5 – Result of natural pouring of Al_2O_3 powder through a funnel

As shown above, the geometric shape that best matches the situation is a cone.
The dimensions formed spontaneously by Al_2O_3 powder cones with mean particle diameter d_{50} = 27 µm are determined based on photographs taken during the tests (i.e. cone diameter, height

and angle of repose). After these tests, the dimensions most suitable for powder cones formed by natural pouring are calculated taking into account a constant volume of 3 cm^3 and a test surface diameter of 4 cm. These dimensions are listed as *Cone H, d* in the table below.

Table 3.4 – Dimensions adopted for alumina powder cones

	Height (mm)	Diameter (mm)	Angle of repose (°)
Cone H, d	12.7	30	40
Cone h, D	8.9	36	26

The angle of repose (cone slope angle with respect to horizontal plane) is then calculated for the five powders using the powder tester method. For powders with mean particle diameter 27 µm ≤ d_{50} ≤ 59 µm, the average angle of repose is 44 degrees, with a variation coefficient of 3%. This is relatively close to the value adopted (i.e. 40 degrees). However, for finer powders with mean diameter d_{50} = 17 µm and 5 µm, the angle of repose is 50 and 58 degrees, respectively. These higher values reflect a stronger adhesion of these powders (due to better cohesion between particles), which suggests a greater resistance to reentrainment.
For the purpose of the experiments, a second set of cone dimensions is also considered (*Cone h,D* in Table 3.4). These dimensions are arbitrarily determined using the same volume as *Cone H,d* and a smaller test surface diameter.

The effect of the shape of the powder deposit on the reentrainment phenomenon will be subsequently studied by varying the shape while maintaining the same volume. Two shapes are chosen:

- The first shape is intended to match that of the flat and multilayer deposits described in the literature. A circular wafer shape is chosen (thickness: 2.7 mm, diameter: 38 mm), which is well suited for the shape of the test surface (also circular).

- The second shape is intended to determine if the presence of corners (as opposed to circular shapes) can modify the airflow properties sufficiently to affect reentrainment. An obelisk shape is chosen (height: 7.4 mm, side lengths: 25 and 15 mm).

Aluminium moulds are used to ensure optimal control of pile shapes and dimensions. The photographs below show the Cone H, d, obelisk- and wafer-shaped powder piles on the test surface after being removed from their respective moulds:

Figure 3.6 – Photographs of pile shapes studied: Cone H, d, Obelisk and Wafer

Once formed, powder deposits are brought directly into the test facility to perform a reentrainment test. Aging-induced deformation of the deposits can therefore be neglected.

Parameter associated with the test surface

The selected parameter associated with the test surface is its roughness, i.e. height of rough spots on the surface where the powder is deposited.

The first step is to choose the material used to manufacture the test surface. The ideal objective is to match the surface conditions of a pipe, and stainless steel is theoretically the best material to do this. However, the slightly magnetic behaviour of stainless steel has been revealed in the course of successive weighing operations. The scale used operates with a bar magnet, and weight measurements are consequently significantly distorted. Considering the high cost of non-magnetic stainless steel, it has been decided that the test surfaces should be made of aluminium.

Four test surfaces with different roughness values have been manufactured:

- test surface with a roughness value of 1.6 µm (conventional roughness),
- polymirror glass-covered test surface (roughness value of less than 0.15 µm),
- two test surfaces with a higher roughness value (6.5 and 12.5 µm).

Airflow-related parameters

The two airflow-related parameters are the airflow velocity (characterized by the near-wall friction velocity v_*) and its initial acceleration.

The variability ranges of these parameters in the BISE test environment were determined in the aeraulics study (Gensdarmes, Roynette, 2011). For reference, in the experiments considered, the air friction velocity parameter can vary from 0.04 m/s to 0.52 m/s (i.e. mean velocity ranging from 0.5 m/s to 10 m/s) and the acceleration parameter can vary from 0.05 m/s^2 to 31 m/s^2. However, as previously noted (Gensdarmes, Roynette, 2011), these two parameters are dependent on one another. The experimental domain used for the test plans must therefore correspond to the overlapping intervals.

According to the literature, the airflow velocity is a major reentrainment factor. This parameter must therefore be varied over a broad range. Consequently, the experimental domain for the air friction velocity is defined as ranging from 0.04 m/s to 0.52 m/s (i.e. from 0.5 m/s to 10 m/s for the nominal airflow velocity in the BISE test facility), corresponding to highly turbulent airflow conditions ($3000 < Re < 60000$). The variability range of the initial acceleration parameter must be reduced, i.e. in order to vary within the same domain as the velocity values, α must only vary between 0.5 m/s^2 and 2.5 m/s^2.

Exposure time

Although recent studies in the literature indicate that the effect of this parameter on particle reentrainment is negligible beyond the first few seconds, it was nevertheless decided to take this parameter into consideration so as to examine the phenomenon in the case of a pile of powder. It should be noted that the test environment used does not allow for reentrainment measurements over very short exposure periods. Therefore, a minimum duration of 2 minutes seems appropriate for the present study.

Fromentin, whose work in 1989 also considered multilayer deposition conditions, notes that the suspended mass stabilizes after an exposure period of 15 to 20 minutes, regardless of initial experimental conditions.

Therefore, as a preliminary approach, it seems that the time interval from 2 to 60 minutes constitutes a satisfactory variability range for studying the effect of the exposure time parameter on the reentrainment phenomenon. Nevertheless, reentrainment measurements over a very short exposure period (30 seconds) will also be performed.

4. Analysis of influential parameters

It is now necessary to examine how the eight parameters considered affect the phenomenon studied. It is recalled that these parameters are powder-related (particle size distribution d_{50}, tap density ρ_{tas}, powder pile dimensions, H_2O content), surface-related (roughness Rug_{SE}), airflow-related (friction velocity v_*, acceleration α) and time-related (Δt).

As a first step, specific test plans are executed with emphasis on "primary" reentrainment parameters, i.e. the most influential parameters among the eight selected. After focusing on these primary parameters, the influence of the other parameters will be examined so as to identify a set of secondary parameters.

4.1. Primary parameters

The first step is to use "screening" test plans to determine the main effects of the parameters considered. As their name suggests, these test plans are used to identify the main parameters affecting the phenomenon studied.

Main effects

Considering the number of parameters to be varied (eight), it is decided to use test plan 12.11p (Plackett-Burman fractioned factorial test matrix), which consists of twelve tests and allows for assessing the influence of up to eleven parameters. However, this test plan requires consideration of two levels of parameters. As discussed in chapter 3, these two levels must be chosen at the minimum and maximum limits (coded variables -1 and +1, respectively) of variability ranges associated with non-interdependent parameters. Table 4.1 shows the test plan used to determine the influence of the various parameters considered.

Table 4.1: Test plan 12.11p (Plackett-Burman test matrix)

Test	d_{50}	Pile shape	ρ_{tas}	H_2O	Rug_{SE}	V	Δt	α
12.11p 1	-1	-1	-1	-1	-1	-1	-1	-1
12.11p 2	1	1	-1	1	1	1	-1	-1
12.11p 3	-1	1	1	-1	1	1	1	-1
12.11p 4	1	-1	1	1	-1	1	1	1
12.11p 5	-1	1	-1	1	1	-1	1	1
12.11p 6	-1	-1	1	-1	1	1	-1	1
12.11p 7	-1	-1	-1	1	-1	1	1	-1
12.11p 8	1	-1	-1	-1	1	-1	1	1
12.11p 9	1	1	-1	-1	-1	1	-1	1
12.11p 10	1	1	1	-1	-1	-1	1	-1
12.11p 11	-1	1	1	1	-1	-1	-1	1
12.11p 12	1	-1	1	1	1	-1	-1	-1
12.11p 13.1… 13.11	0	0	0	0*	0*	0	0	0

where d_{50} : Mean particle diameter of Al_2O_3 powder used
Pile shape : Dimensions of Cone h, D (-1) and Cone H, d (+1), with volume = 3 cm^3 and
- Height h : 8.9 mm and H=12.7 mm, respectively
- Diameter D : 36 mm and d = 30 mm, respectively

ρ_{tas} : Tap density of powder,
H_2O : H_2O content of powder,
Rug_{SE} : Roughness of test surface,
V : Mean airflow velocity,
Δt : Duration of exposure to airflow,
α : Initial airflow acceleration.

The experimental domains of the parameters chosen for test plan 12.11p are determined with a view to obtaining the largest possible variability ranges. Furthermore, the parameters are independent of one another throughout the domains considered (indispensable test plan requirement). The variability ranges adopted for this first test plan are listed in the table 4.2 below.

* Mean condition set to 1 for technical reasons.

Table 4.2 – Experimental domains of 8 parameters considered for test plan 12.11p (Plackett-Burman test matrix) – Al_2O_3 powder – Volume: 3 cm^3

Parameters	Experimental domain		
	-1	***0***	***1***
Mean particle diameter d_{50} (µm)	5	27	59
Tap density ρ_{tas} (g/cm^3)	1.5	1.75	2
H_2O content (mgH_20/g)	0	1*	1
Cone height and diameter (mm)	h = 8.9 D = 36	H = 12.7* d = 30*	H = 12.7 d = 30
Test surface roughness Rug_{SE} (µm)	Polymirror	1.6*	1.6
Air friction velocity v_* (V) (m/s)	0.04 (0.5)	0.29 (5)	0.52 (10)
Exposure time Δt (min)	2	15	60
Initial acceleration α (m/s^2)	0.5	1.5	2.5

*Values for which mean conditions have not been experimentally determined and for which coded variable 1 has been arbitrarily assigned.

Each test is performed once. However, repetitions are performed at the centre of the experimental domain, i.e. for mean values (coded variable 0) of parameter variability ranges. These repetitions at the centre of the experimental domain are used to estimate the dispersion of test results over the entire domain, provided that the assumption of homoscedasticity is valid. This assumption considers that standard deviations are constant throughout the experimental domain. For the purpose of this study, it is initially postulated that this assumption is valid for the phenomenon considered.

Specific analysis of airflow velocity and particle size distribution parameters

In order to study only the influence of the airflow velocity and particle size distribution parameters on the resuspended fraction, the other six (non-significant) parameters are set to mean values representative of the variability ranges previously established (see Table 4.2), i.e.

- H_2O content: H_2O ~ 1 mgH_2O/g
- Cone dimensions: H = 12.9 mm and d = 30 mm
- Density: ρ_{tas} = 1.75 g/cm^3
- Exposure time: Δt = 15 min
- Acceleration: α = 1.5 m/s^2
- Surface roughness: Rug_{SE} = 1.6 µm

For reference, the experimental domains of the two primary parameters are:

- Velocity: $0.04 \leq v_* \leq 0.52$ m/s ($0.5 \leq V \leq 10$ m/s)
- Particle size: $5 \leq d_{50} \leq 59$ µm

Airflow velocity threshold

Given the extent of the experimental domains for the velocity and particle size parameters, it is essential to optimize the number of tests to be performed. Therefore, the first step is to identify the

parameter regions to be explored in detail by executing a second test plan (13.2p) shown in the table 4.3 below.

Table 4.3: Test plan 13.2p (Central composite test matrix)

Test	d_{50}	V
13.2p 1	-1	-1
13.2p 2	1	-1
13.2p 3	-1	1
13.2p 4	1	1
13.2p 5	$-\beta^{**}$	0
13.2p 6	β^{**}	0
13.2p 7	0	$-\beta^{**}$
13.2p 8	0	β^{**}
13.2p 9.1…9.4	0	0

where d_{50} = Mean particle diameter of Al_2O_3 powder used
and V = Mean airflow velocity

The six predefined parameter values for this test plan are:

Pile shape:	Cone H,d
Tap density of powder:	ρ_{tas} = 1.75 g/cm^3
H_2O content of powder:	H_2O = ~1mgH_2O/g
Roughness of test surface (µm):	Rug_{SE} = 1.6 µm
Duration of exposure to airflow:	Δt = 15 min
Airflow acceleration:	α = 1.5m/s^2

A sequential test plan (2^n where *n* is the number of tests) can be developed by varying the parameters around a circle centred at 0 (mean value) and with radius β.
In order to visualize the position of the tests within the experimental domain, Figure 4.1 shows test matrix 13.2p (represented by squares) as a function of the coded variables assigned to the velocity and particle size parameters. Tests performed for test plan 12.11p are also shown, represented by circles.

** $\beta = \sqrt[4]{2^{n'}}$ where n' is the number of quantitative factors considered in the test plan (here n' = 2, i.e. β = 1.4)

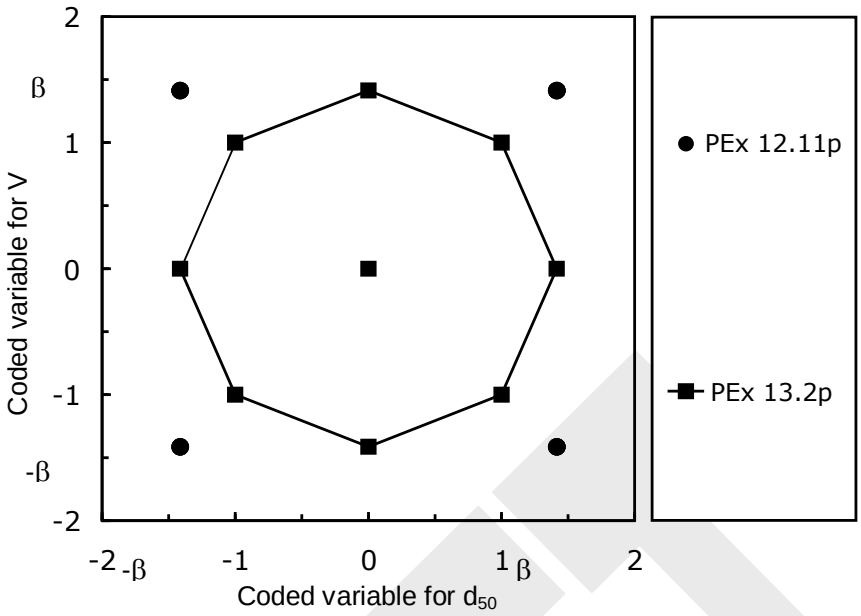

Figure 4.1 – Position of test points in test matrices 12.11p and 13.2p

The above figure shows that by carefully assigning values of d_{50} and V (and therefore v_*) to the coded variables, test matrix 13.2p can be used to scan the entire experimental domain. In this manner, it is possible to supplement the results of test plan 12.11p located at the four ends of the experimental domain so as to perform a five-level analysis of the two primary parameters using only nine tests.
The five velocity and particle size values adopted for test plan 13.2p are shown in Table 4.4. As previously explained, the coded variables of test plan 12.11p (-1, 0 and 1) are associated with the limit and mean values (-β, 0 and β) of the new test plan (i.e. -1.4; 0 and 1.4).

Table 4.4 – Particle size and velocity values chosen for specific analysis with respect to K_R

Parameter level	**-β**	**-1**	**0**	**1**	**β**
d_{50} *(µm)*	5	17	27	46	59
v_* *(m/s)*	0,04	0,13	0,29	0,45	0,52
(V) (m/s)	(0,5)	(2)	(5)	(8,5)	(10)

Six tests will be repeated at the centre of the experimental domain (coded variable 0), taking into account the dispersion of results over the entire domain by once again postulating the assumption of homoscedasticity.

5. Conclusion

For the purpose of the study, eight parameters to be varied have been selected (out of a total of 20 parameters). Using the test plans previously established (amounting to approximately 40 tests), it will be possible to identify a set of primary parameters significantly affecting particle resuspension. Once these primary parameters have been identified, a new series of experiments will need to be conducted by assigning predefined values to these parameters so as to identify a set of secondary parameters. The influence of the shape of the powder deposit will then be studied independently of the other parameters.

6. References

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