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Influence of the amount of fine particles on rheological properties of uranium dioxide powders

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Abstract

In industry, the use of powders is very common. Understanding granular flow in silos, hoppers or pneumatic conduits is necessary. This study investigates the characterization of the rheological properties of uranium oxide powders in the context of a powder metallurgy process for nuclear fuel pellets manufacturing. The flow and arrangement properties of these powders must be compatible with a reproducible feed of press moulds. This may require a modification of the physical characteristics of the powders. In particular, particle size is known to have a great impact on the rheological properties and a significant effect of fine particles (less than 100 µm) is suspected. In this study, we have quantified this influence of particle size distribution on rheological properties of UO_2 powders, especially the influence of the amount of fine particles. Two complementary approaches have been used. The first approach is to characterize the powder in a static state: the density, compressibility and shear measurements make it possible to understand the behaviour of the powder when it is moved from static state to dynamic state. The second approach is to characterize the behaviour of the powder in the dynamic state. It allows understanding the flow capacity of a powder in the process. To answer the problem of reproducible filling of the press moulds, it is essential to characterize the two regimes, that is to say the flow of the powder from a hopper or a shoe, then the behaviour in the dynamic state. The amount of fine particles has been more especially varied because the cohesive forces are considered predominant with respect to gravitational forces for particles smaller than 100 µm. A threshold of the rate of fines has been demonstrated in both types of measurements. Beyond this threshold, the behaviour of the powder is approximatively the same.

Keywords: uranium dioxide, powder, particle size distribution, fines, rheology, filling

I. Introduction

The sodium-cooled ASTRID nuclear reactor under study will use a different fuel than pressurized water reactors. This fuel has particular characteristics which require a suitable manufacturing process. The current reference powder at the entrance of the pressing step does not have properties allowing a reproducible filling of press moulds. It induces the necessity of grinding the pellets after sintering. This rectification of sintered pellets is costly and also radiologically penalizing because of the generation of radioactive dust. The aim of this study is to give orientations for a suitable

modification of the characteristics of powders, in order to have a reproducible filling of press moulds and avoid grinding.

Fu [1] investigated the effect of particle shape and size on the flow and consolidation of lactose powders. His results indicate that the shape and size of the powder particles both significantly affect the flowability. The influence of particle size on flowability is also described in many publications [2]–[4]. Following Li [2], granular materials can be classified into two categories: powders smaller than 100 µm (called fines) and those larger than 100 µm. A strong influence of the fine particles on powder behaviour is suspected. Firstly, cohesion forces (Van der Waals forces) are considered predominant in these fines [2]. Secondly, fines have a strong impact on the reorganization of the powder bed and can cause a stacking defect [5]. The fines can adsorb to the surface of larger particles increasing frictional forces and thus obtaining a poor flow [6]. In other cases, the fines may coat coarse particles forming a smooth surface. They therefore reduce frictional forces and facilitates the flow [6].

A preliminary study was carried out on three granulometric classes of UO_2 granules [7]: 0-100 µm, 100-200 µm and 200-300 µm. It has been shown that the population of fine particles (<100 µm) corresponds to a very cohesive powder, with a poor flow. In this study, we consider a powder with a broad particle size distribution and we search the acceptable amount of fines to obtain a "good" filling. A "good" filling is a homogeneous and reproducible filling. For that, understand the behaviour of our powder is necessary.

There are different methods for characterizing the flow properties of a powder for a “good” filling [1]. The powder can be in static or dynamic state. To understand rheological properties, it is necessary to observe how powder moves from a static mode to a dynamic system, and thereafter its behaviour in dynamic mode. Methods in the first category are for example density measurement, compressibility and shear test. The latter makes it possible to obtain precise results and to characterize the powders in their industrial context [8]. It thus makes it possible to reproduce the constraints applied on the powder in its industrial application. A second family of methods characterizes the powder flow and the filling behaviour: dynamic rheometer measurements, permeability and aeration measurements.

Leturia [9] compared the traditional methods of powder flow characterization with rheometer and shear cell measurements. He concluded that the choice of the powder characterization method depends on the process conditions. The flow of a powder cannot be characterized by a single index but by a combination of different tests.

In this study, the influence of the quantity of fine particles on UO_2 powder mixtures will be presented. A direct comparison between the different UO_2 powder flow characterization indices is done as a function of the quantity of fines present in the mixture in order to understand the phenomenon occurring within the powder in the context of a powder metallurgy process for nuclear fuel manufacturing, especially for the filling of moulds.

II. Materials and methods

1. Sample preparation

The raw powder was firstly compressed at 150 MPa to make granules (Figure 1a). This pressure makes it possible to obtain granules that are strong enough to be handled and studied but that can be broken easily during pressing, to have no effect on the final microstructure. The UO_2 powders are handled in a glove box (Figure 1b). A sieve grinder (Freewitt type OxcilloWitt-Lab) was used to perform grinding-forcing in a reproducible manner. Granules can then be made reproducibly with little operator effect. This granulation allows a good repeatability in the manufacturing of our samples and leads to granules that can be considered as roughly representative of dense agglomerates obtained after a preliminary milling step as expected in the current reference process. Reproducibility and repeatability has been demonstrated via measurements of particle size distributions with a dry granulometer (Malvern Mastersizer 3000).

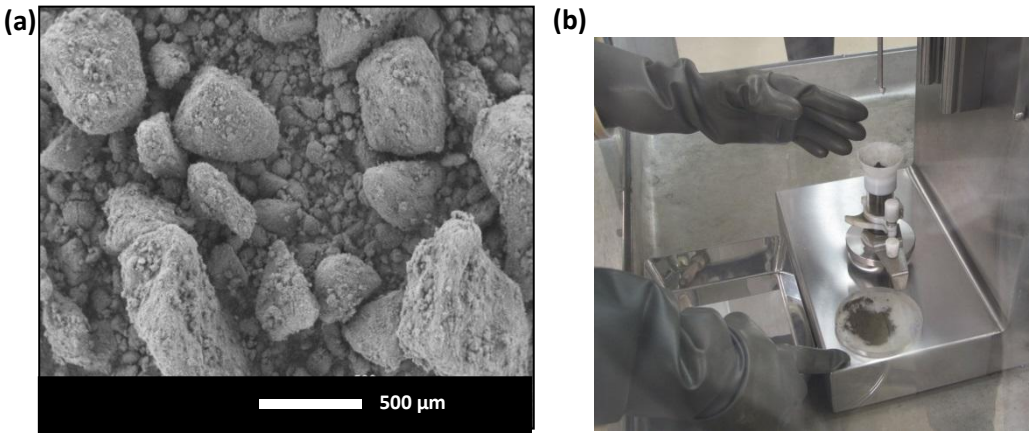


Figure 1 : SEM image (SE Mode), uranium dioxide powder (a) and powder handling in a glove box (b).

The maximum size of granules is about 550 µm. Fine particles are separated by sieving and reintroduced to the population of large particles to obtain several calibrated mixtures.

One sample contains about 200 g. Five samples were prepared with different amounts of fines: 0/10/30/50 and 100 wt% of fines (Figure 2). The mixtures were done using a turbula mixer for 10 min at a speed of 23 min^{-1}.

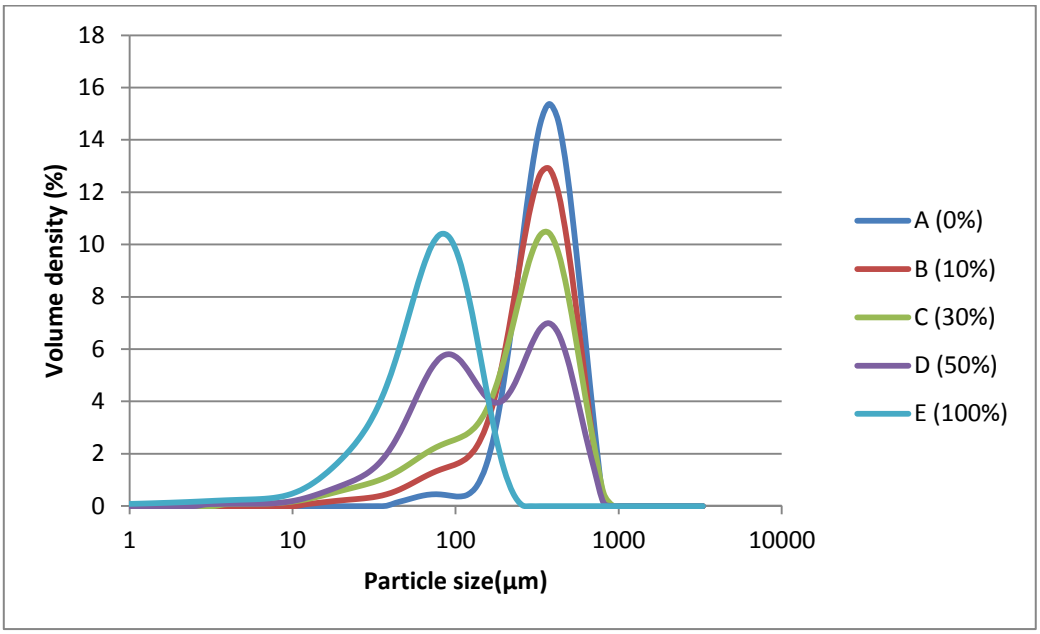


Figure 2 : Different particle size distributions (volume fraction).

The quantity of UO_2 powder available is very limited. Despite this each sample was repeated 2 times. And the extreme samples (A and E) containing respectively 0 and 100% will be made 3 times each. For each type of measurement, an absolute uncertainty was determined based on the measurements made on the extremes. The error calculation will be done on these two extreme samples with the Student test for a 95% confidence interval.

The diameters Dv10, Dv50 and Dv90 are the diameters for which respectively 10%, 50% and 90% of the volume of the population is below these diameters values of these characteristic diameters are given in Table 1.

Table 1 : Values of characteristic diameters.

Sample	Ratio of fines (%)	Dv (10) (µm)	Dv (50) (µm)	Dv (90) (µm)
A	0	206	358	566
B	10	115	317	537
C	30	59	279	527
D	50	42	156	476
E	100	23	70	136

2. Density measurements

The aptitude for the arrangement of a powder is linked to its cohesion and flowability. If a powder is gently poured into a container, it will have a loose organization. Under the effect of numerous vertical shocks, the powder will settle changing the organization of the initial powder until a compact

reorganization. This powder's rearrangement is due to the evacuation of the interstitial air. The use of a volumenometer allows this transition from an aerated density ρ_0 to a packed density ρ_t following a standard protocol according to the European Pharmacopoeia standard.

From the measurement of aerated densities and packed densities, the Hausner index [10] can be calculated:

$$Hausner\ Index = \frac{\rho_t}{\rho_0} \qquad \text{Equation 1}$$

Depending on the Hausner index value, the powder flowability can be predicted qualitatively (Table 2) [11].

Table 2 : Flowability as function of Hausner index.

Flowability	Hausner index
Excellent	1,00 - 1,11
Correct	1,12 - 1,18
Reasonable	1,19 - 1,25
Fair	1,26 - 1,34
Poor	1,35 - 1,45
Very poor	1,46 - 1,59
Extremely poor	> 1,60

3. FT4 Rheometer

Freeman Technology's FT4 rheometer uses several characterisation tests to understand the flow of powders [12].

3.1. VFR test (Variable Flow Rate test)

The principle of the test is to introduce a powder sample into a cylindrical cell in which a specific blade rotates and moves vertically along a helical path. The blade can move at different speeds. In this study, we have used a cell of 10 mL and a blade of 25 mm in diameter.

Different indices were determined to evaluate the flow properties of the powder [9]. For each test, 11 measurements are made:

- A series of 7 tests repeated at the same rotational speed of the blade (100 mm/s). The value obtained at the last test is used to determine the energy necessary to move a given volume of powder, called BFE (Basic Flow Energy). The Normalized Basic Flow Energy (NBFE) it also used to compare powders [9], taking into account the different sample masses.

$$NBFE = \frac{BFE}{sample\ mass} \qquad \text{Equation 2}$$

- The results obtained also make it possible to define a stability index, noted SI (Stability Index). This index is the ratio of the total energies during the last and the first tests.

- Then another series of 4 tests at different rotational speeds of the blade -100, -70, -40 and -10 mm/s are realized. This reduction in the rotational speed of the blade by a factor of 10 is used to determine the impact of the flow speed on the total energy. A flow index, noted FRI (Flow Rate Index), makes it possible to quantify this effect by comparing the values obtained for the maximum and minimum speeds.

- Before each measurement, a conditioning of the powder is carried out. The powder is then put in a reproducible and unconsolidated state. During the moving up of the blade, the energy needed to move the powder is then called the specific energy SE.

3.2. Compressibility

In the FT4 device [9], the compressibility of the powder describes the ability of a bulk powder's volume reduction when it is slowly compressed under a given normal force. The powder is poured into a cylindrical cell with a diameter of 25 mm and a volume of 10 mL. A porous compression piston of 23.5 mm diameter is applied on the powder. The tests are carried out for pressures ranging from 1 kPa to 15 kPa.

3.3. Permeability

The permeability measurement permits to evaluate the capacity of a powder bed to allow the air to pass at different consolidation stresses for a constant speed. For the test, a 25 mm diameter container with a volume of 10 mL is used, coupled to a porous aeration base connected to an aeration module. First of all a conditioning of the powder is carried out, then a normal stress varying between 1 and 15 kPa is applied on the top of the powder bed. A constant air flow rate of 2 mm/s passes through the powder bed continuously. The pressure drop in the powder bed is then measured as a function of the normal stress applied.

The permeability of the bed is :

$$k = \frac{q.\mu.L}{\Delta P} \qquad \text{Equation 3}$$

With k permeability (cm²), q superficial velocity (cm/s), μ air viscosity (Pa.s), L length of powder bed (cm), ΔP pressure drop across the powder bed (mbar)**.**

4. Shear cell

Jenike [13] has developed a method based on the measurement of the shear strength of the material. The shear cell measures the variation in shear stress required to set the consolidated powder bed into motion at different normal stresses. Bell [14] confirms the validity and reproducibility of this method for different powders.

During a shear test, two stages can be distinguished: the pre-shearing step followed by the shearing step. The pre-shearing step is necessary before each shear measurement. It makes it possible to repeat the measurements under the same conditions and to erase the powder hysteresis. The pre-shearing step consists of applying a known normal stress σ_p to the powder, and then shearing the powder until it reaches a constant density.

During the shearing step, a normal stress **σ** lower than the normal pre-shear stress (σ_p) is applied, then a shear stress **τ** is applied until the powder bed breaks. We obtain several couples (τ, σ) to trace the yield locus (Figure 3a).

The yield locus is supposed in linear form according to the Mohr-Coulomb failure criterion [15]:

$$\tau = \mu.\sigma + c \qquad \text{Equation 4}$$

Thus, we can determine the following parameters [8], [16]:

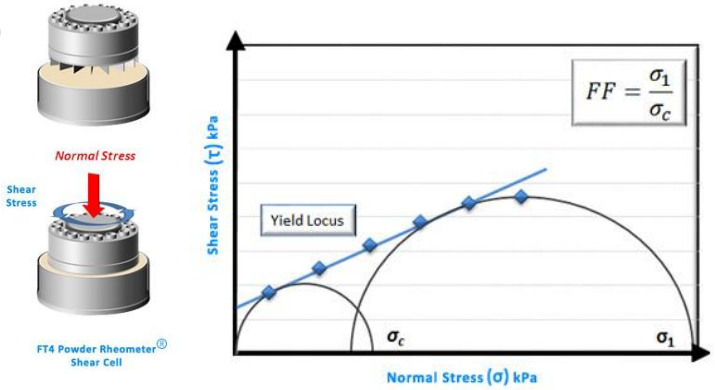
- The Major Principal Stress (MPS) $\boldsymbol{\sigma_1}$ obtained by drawing the large Mohr circle: it characterizes the maximum stress that could have been applied on the powder.
- The Unconfined Yield Strength (UYS) $\boldsymbol{\sigma_c}$ obtained by drawing the small Mohr circle: it characterizes the resistance of the powder subjected to a simple compression.
- Mohr circle **μ** is the internal coefficient of friction: μ = tan (ϕ). ϕ is the angle of inclination of the yield locus with the horizontal axis, also called the internal friction angle.
- At σ = 0, the cohesion **c** of the powder is obtained. This point corresponds to the initial cohesion of the powder without any constraint being applied on it.

The flowability factor is defined as:

$$FF = \frac{\sigma_1}{\sigma_c} \qquad \text{Equation 5}$$

It is a first approach of the flow behaviour (Figure 3b).

(a)



(b)

Flowability Factor FF	Flow behaviour
FF < 1	Not flowing
1 < FF < 2	Very cohesive
2 < FF < 4	Cohesive
4 < FF < 10	Easy-flowing
10 < FF	Free-flowing

Figure 3 : Principe shear test and yield locus (a); classification of flowability factor FF (b) [16] .

After having measured several yield loci (thus at different pre-shear σ_p), several pairs ($\boldsymbol{\sigma_c}$, $\boldsymbol{\sigma_1}$) are obtained. The curve passing through the different couples represents the flow function. According to Jenike [13], the flow properties of a powder can be determined by its flow function.

The FT4 rheometer can be equipped with shear cell with different volumes [12]. A 10 mL shear cell is used for our study. The choice of this cell results from an optimization with respect to the quality of the results and the quantity of powder involved [7].

III. Results and discussion.

1. Evaluation of the powder transition from static to dynamic state

1.1 Density and compressibility

The density test allows understanding the rearrangement of the grains without applying a stress. On the contrary, for the compressibility test the rearrangement of grains is done by applying a normal stress. These two tests are not a direct measurement of the flow properties but allow predicting qualitatively the behaviour of the powder during the flow. Results are given in Table 3.

The FT4 can also measure the aerated density (called conditioned bulk density CBD). The results are strongly affected by the initial filling of the powder by the operator. However as shown in Table 3, the aerated density measured with both methods is similar which may lead to the conclusion that the operator effect is then low.

$$Conditioned\ Bulk\ Density\ (CBD) = \frac{Split\ Mass\ after\ Conditioning}{Sample\ Volume} \qquad \text{Equation 6}$$

Table 3 : Density measurements and compressibility (FT4).

Sample	Ratio of fine particles (%)	aerated density (g/cm3)	tapped density	Hausner ratio	compressibility FT4 at 15 kPa (%)	CBD (FT4)
A	0	2,8	3,3	1,20	5,6	2,74
B	10	2,7	3,3	1,23	7,0	2,78
C	30	2,5	3,3	1,36	9,8	2,40
D	50	2,5	3,5	1,39	15,0	2,27
E	100	2,1	2,9	1,41	21,3	2,08

Figure 4 shows that the powder with 100 % of fines has the highest compressibility values (FT4) at all applied stresses compared to other powders. The difference in volume is all the more high as the level of fines is high (Figure 4). We can assume that for higher fines content, more air is trapped in the powder bed and therefore the powder has a high compressibility.

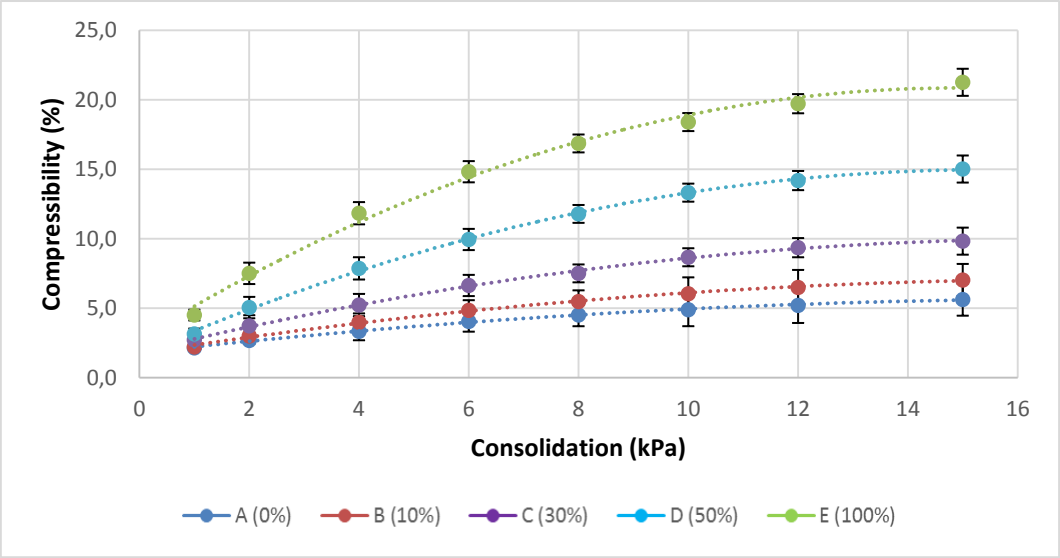


Figure 4 : Compressibility as function of consolidation.

The Hausner Ratio follows the same tendency as compressibility. The flowability qualitatively estimated from Hausner ratio seems to decrease when the fines content increases. However, whereas a linear correlation can be observed between the compressibility and the quantity of fines (Figure 5), for the Hausner ratio we observe the appearance of a threshold of fines above which the behaviour of the powder changes very little.

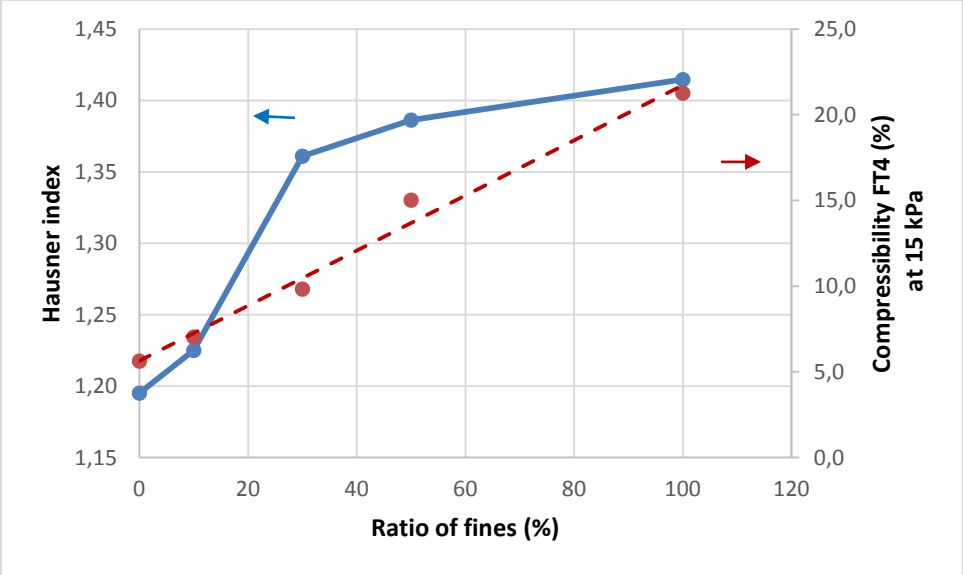


Figure 5 : Hausner index (blue) and compressibility (red) FT4 versus quantity of fines.

For each powder, the compressibility test was carried out three times successively in order to observe whether it modifies the characteristics of the powder or not. In our case, for a same sample the compressibility does not vary. This is why the compressibility test, simple and fast, will be used before and after each test of this study, as a reference test. It will allow following the evolution of the

behaviour of the powder during the characterization of the flow of the powder and detecting if the powder changes (break of agglomerates, etc.).

1.2 Shear test

Shearing allows to characterize the complexity of the powder during its transition from the static state to the dynamic state when for example opening the bottom of a hopper [17].

1.2.1 Flow function.

The shear test is applied for four values of pre-shear stress for each powder: 3, 6, 9 and 15 kPa [18]. One sample is used for the entire shear test. The tests are therefore done in increasing pre-shear stresses. To verify that the powder was not modified by all these tests, a last shear test at a stress of 3 kPa is performed and compared with the first test at 3 kPa. For all of our samples, the powder did not change due to shearing.

The shear test results were used to plot the flow functions for each sample (Figure 6) [8]. Samples A and B, consisting of 0 and 10% fines respectively, have better flow functions than the other samples.

Because of their size, fine particles have a larger specific surface area than larger particles. This is possibly why, starting from a certain threshold of fine particles, they dominate the behaviour of the mixture. This could explain the similarity between the samples C (30%), D (50%) and E (100%). The FF cannot be differentiated between samples with fines content greater than 30% (Figure 7).

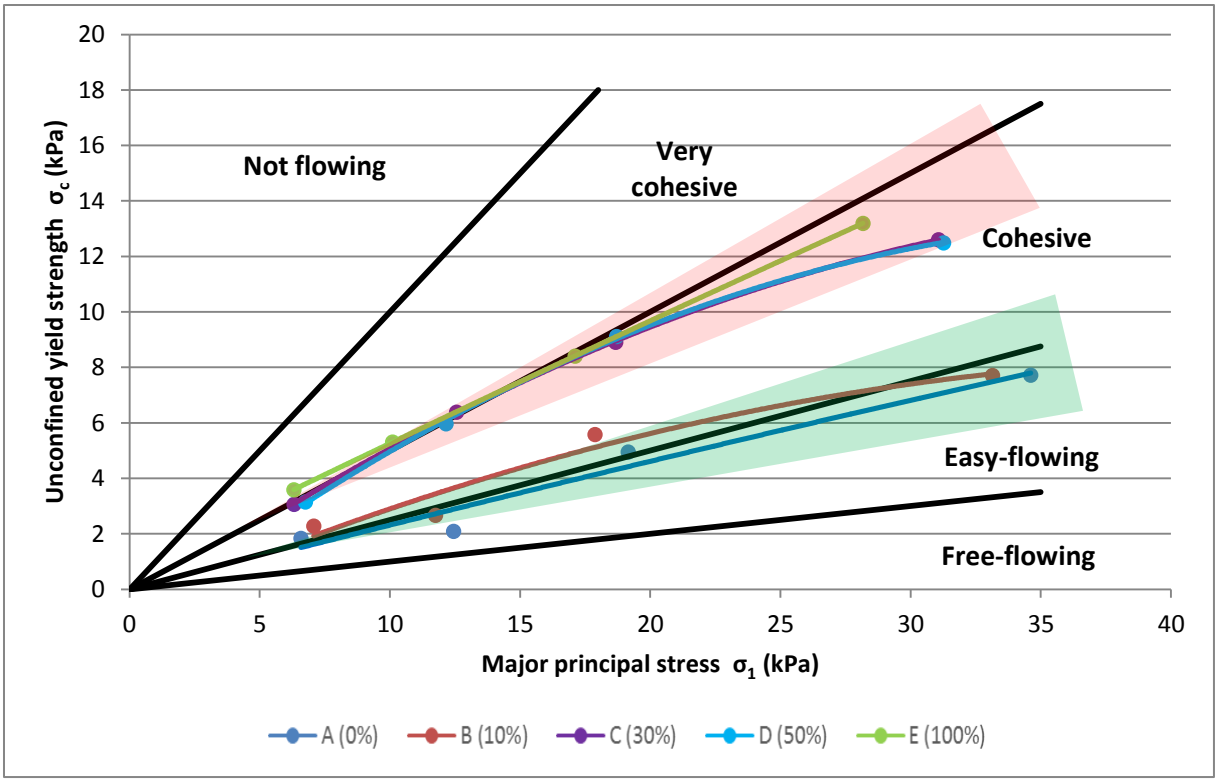


Figure 6 : Flow functions for the various particle size distributions.

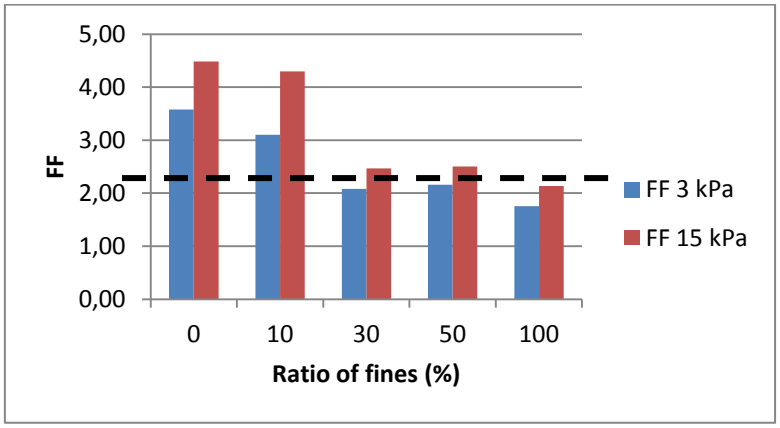


Figure 7 : Variation of the Flow factor FF as function of quantity of fines.

1.2.2 Cohesion

Cohesion was also measured during the shear test. The cohesion of the powder is plotted as a function of the quantity of fines (Figure 8). The sample with only fines (E 100% fines) is cohesive compared to the sample without fines (A 0% fines).

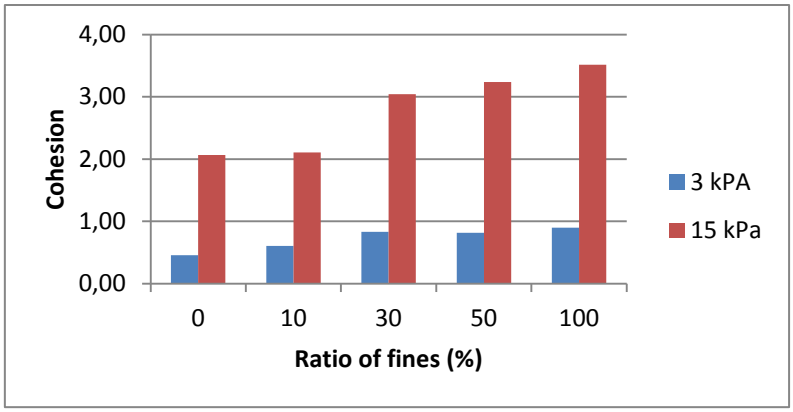


Figure 8 : Evolution of cohesion as function of quantity of fines (for pre-consolidation stresses 3 kPa and 15 kPa).

Cohesion is about the same for 0 and 10% of fines. It is higher for 30% of fines and it remains about the same above this value of 30%, up to 100% of fines. So the cohesion criterion cannot differentiate the samples with amounts of fines higher than 30%.

2. Evaluation of the powder in dynamic state

2.1 Permeability test

When filling the mould with the powder, it is necessary to evacuate the air trapped in the mould. The permeability test is a characterisation of the filling ability because it gives an indication of the ease of the air, trapped in the mould, to cross the bed of powder to allow a regular flow of the powder for a “good” filling.

The pressure drop (DP) and the permeability k (equation 4) are plotted as a function of the consolidation stress applied on the powder bed (Figure 9).

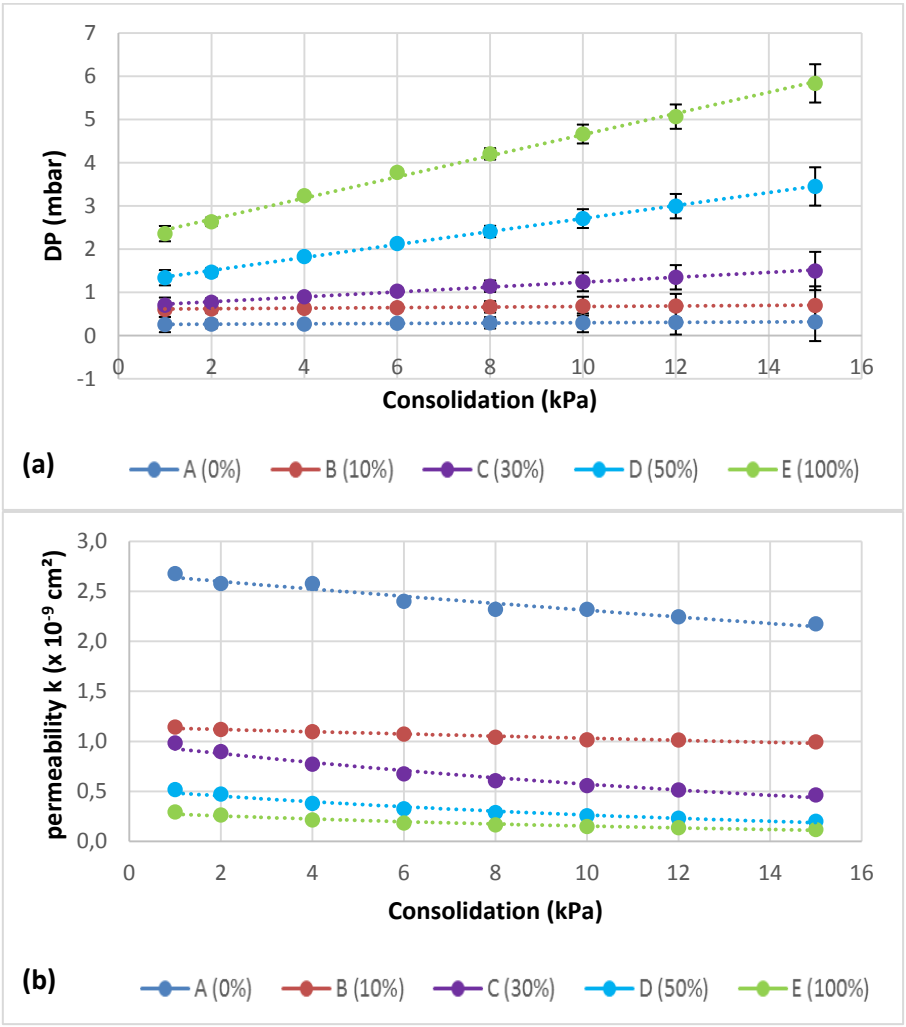


Figure 9 : Evolution of DP (a) and permeability k (b) as function of consolidation for different quantities of fines.

The pressure drop is a linear function of the consolidation stress for each sample. It is high for powders with a high rate of fines. The fines cause the increase of the pressure drop because they lower the interstitial space between particles. As a result, the pressure drop within the powder bed E (100% fines) is the highest.

The permeability of the powder can then be determined as a function of consolidation. As seen previously, the permeability is inversely proportional to the pressure drop. As the quantity of fines increases, the pressure loss increases and therefore the permeability k decreases (Figure 10).

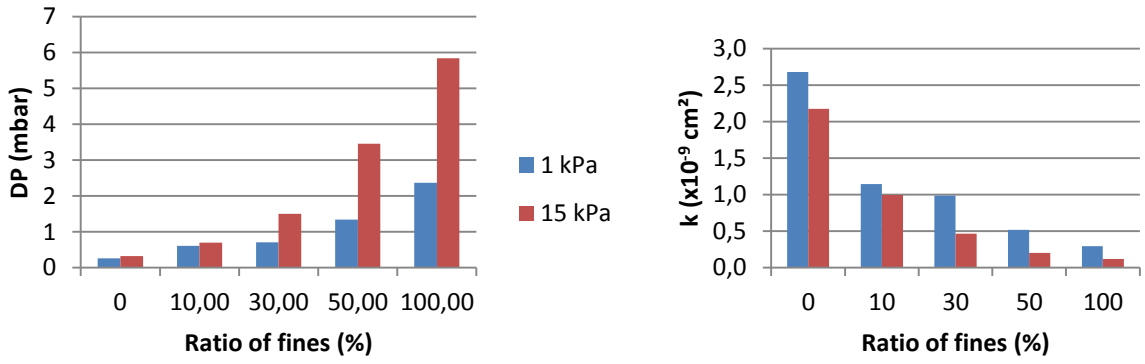


Figure 10 : Pressure drop DP (left) and permeability k (right) versus quantity of fines (for 1 kPa and 15 kPa).

The permeability is linked to the volume and structure of the inter-particulate porosity. When the quantity of fines is zero (sample A), a high permeability is observed. As soon as the fines are introduced into the mixture, they occupy the inter-particle spaces: the air passes through the powder bed with difficulty, which explains a sharp drop in permeability [12]. Because of this, the fines play strongly on the flow of the powder in a mould and therefore on the ability to obtain a reproducible and stable filling. This test can differentiate all our powders, even those whose quantity of fines is greater than 30%, unlike shearing. The fines may have a critical role on the filling according to the criterion of air evacuation.

2.2 Dynamic tests

To obtain a deeper understanding of the flow of the powder, dynamic tests were performed on our 5 samples (VFR tests). The results of BFE tests are shown on Figure 11, as well as the characteristic indexes of the mixtures.

The basic flowability energy (BFE) measured is higher for A and B samples (0 and 10% of fines) than for C, D and E samples (30, 50 and 100% of fines). As this test is considered representative of the free flow of the powder in a confined environment, this result could mean that the free flow of the powder is more difficult for the small amounts of fines. Indeed, if the BFE is small, the energy required to flush the powder is low and the powder is supposed to have a good flow and to be non-cohesive. On the contrary, if the BFE is high, the powder is considered cohesive.

However this theory is not always true, when the blade crosses a cohesive powder formed of small particles (for example E 100% of fines), the blade creates a channel inside the powder bed. Fines particles do not spontaneously fill the void let by the blade, so it moves a small volume and the energy measure is not representative of the energy necessary to move the bed.

In the same way as for the BFE, the SE is high for powders A and B compared to the other samples (Figure 11).

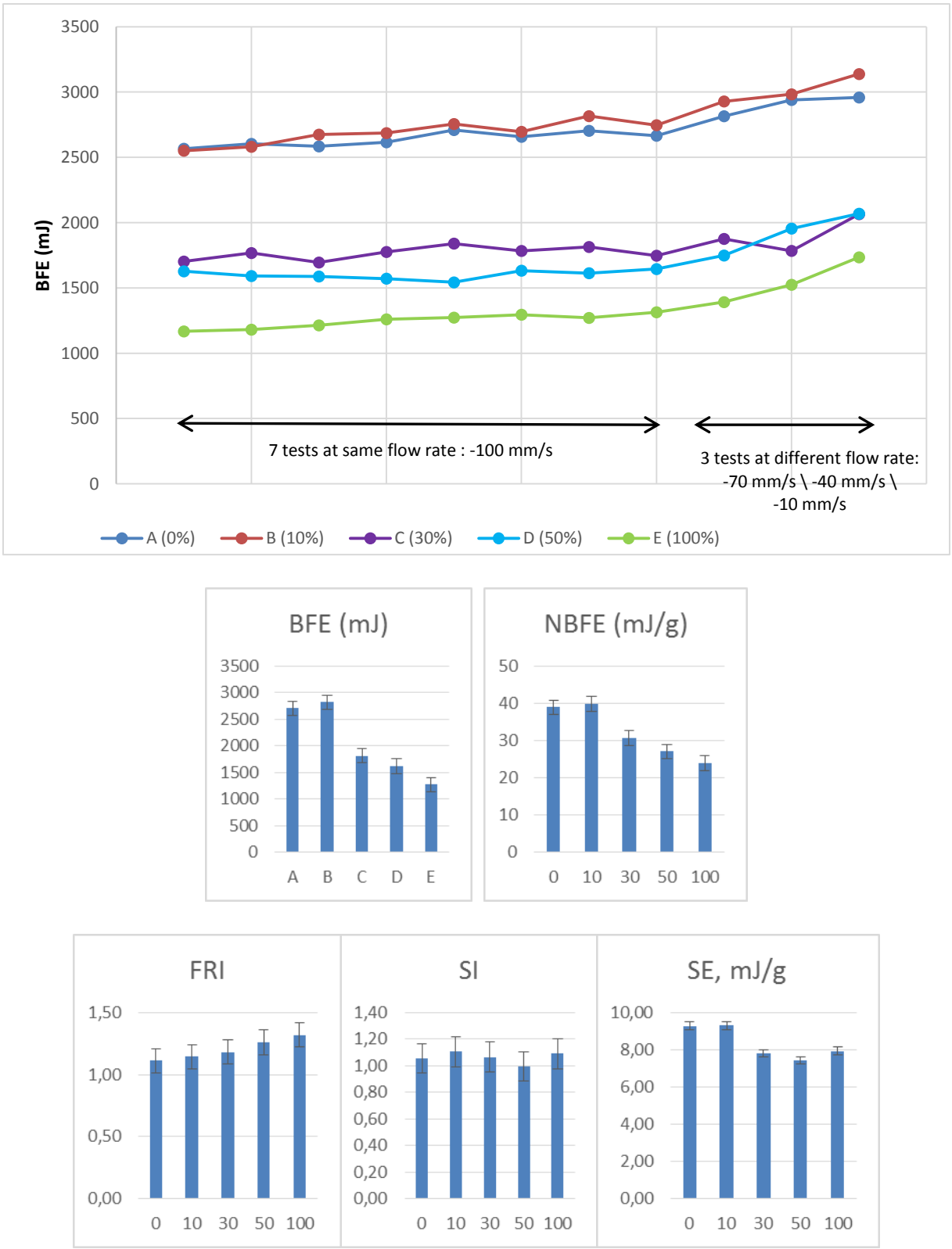


Figure 11 : VFR tests and the different indexes as function of quantity of fines (BFE, NBFE, FRI, SI and SE).

When the speed of the blade decreases, highly compressible powders can less easily form a channel that facilitates the movement of the blade. It is why the FRI increases with the quantity of fines

(Figure 11). Li [19] has tried to understand these phenomena that contradict the BFE. He observed the flow of the powder during the test: when the powder is cohesive, the blade lets a gap behind it unlike non-cohesive powders; the powder flows directly after the blade crossed it. To understand the phenomenon, he set up a "verification test". This test consists of rotating the blade only at the surface of the powder bed at different speeds. A "block of powder" consisting of several agglomerates of different sizes is formed on the blade and will fall gradually. This is the phenomenon of "build-up / collapse" [19]. This sudden drop and this formation of continuous agglomerates cause oscillations of the measured torque. These oscillations are much more significant for cohesive powders.

In our case the VFR test is considered destructive. After undergoing this test, the powder no longer has the same properties as in its initial state. This may be due to the destruction of a part of the particles; the blade breaks some agglomerates to make fines. The compressibility test is a reference way to understand what happened during the VFR test. If we take extremes samples A (0% fines) and E (100% fines), the difference in compressibility is very marked before and after VFR (Figure 12). Moreover the measurement of the particle size distribution before and after VFR made it possible to confirm this phenomenon of fines formation.

During the VFR test, the SI does not discriminate the different powders (Figure 11), its value is about 1. The compressibility test shows the formation of fines. The SI is still stable because the quantity of fines formed has no influence on the VFR test. That's why the NBFE does not vary throughout the test. This index does not have a great signification for our powders.

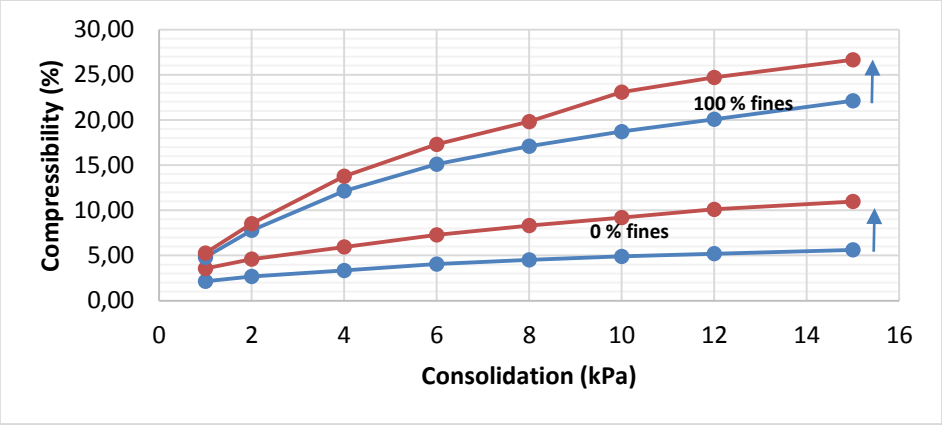


Figure 12 : Compressibility test before and after the VFR test (For 0% and 100% of fines).

3. Comparison shear test versus permeability tests.

The flow factor (FF) is representative of the passage of powder from the static state to the dynamic state. While the permeability makes it possible to understand the flow capacity of the powder for filling. We try to identify families according to the two regimes of the powder: the setting in flow and the flow.

The FF is plotted as a function of the permeability (Figure 13). The more permeable a powder is, the more it allows the air to pass easily. This is important for a reproducible and homogeneous filling of press moulds.

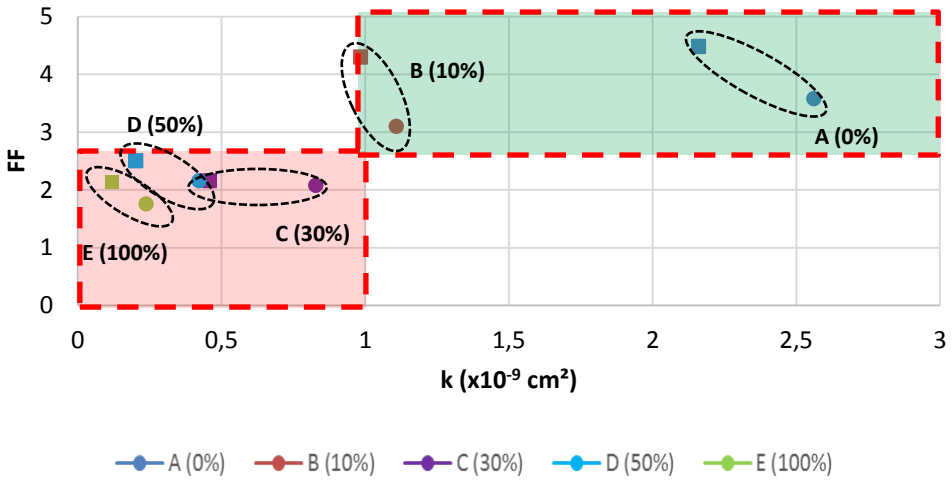


Figure 13 : Variation of Flow Rate FF as a function of permeability for different mixtures [air flow=2mm/s] (circle: 1 kPa / square: 15 kPa)

The results show two families:

- Cohesive zone with FF < 2.5 and k < 1 (with air flow of 2 mm/s): for samples C, D and E (the quantity of fines is greater than 30%). We are in the "very cohesive" powder region. The powder does not allow air to pass and trap it. The powder is very cohesive and therefore flows with difficulty.
- Non-cohesive zone FF > 2.5 and k > 1 (with air flow of 2 mm/s): For the samples A and B (0 and 10% of fines), the compressibility varies little, there is a little increase of the cohesion (between 0.5 and 2 kPa) and the powder is very porous. The powder has a better flow than the powders of previous zone.

This confirms that from a certain threshold of fines between 10 and 30 %, the powder adopts an identical behaviour "very cohesive".

IV. Conclusion

Various methods make it possible to characterize the flow of powders. The characterization is done at two levels:

- Characterize the powder transition from the static to dynamic state: density, compressibility and shear measurements make it possible to understand the behaviour of a powder when it begins to flow. Shear tests have demonstrated an increase in cohesion with the increase of fines in our mixture. This is not surprising because cohesion forces are supposed to be predominant

over gravitational forces for particles smaller than 100 µm[2]. A threshold of fines between 10 - 30% has been determined. Beyond this threshold, the powder has the same flow behaviour.

- Characterize the behaviour of the powder in dynamic state allows to account especially for the filling capacity of the powder. The permeability tests and the dynamic tests permit to observe the influence of fines on the capacity to evacuate the air and also on the energy needed to move the powder. If a powder contains more fines, it traps easily the air and thus leads to a “bad” behaviour during filling. So when the amount of fines increase, the flow is degraded and the filling of the press moulds may become non homogenous and non-reproducible.

It is difficult to find direct links between the two systems (static to dynamic/dynamic). The combination of these two tests can often present contradictory results. To predict filling behaviour, it is essential to characterize powders in both regimes [20]. To better know the behaviour of our powders in the process, it is necessary to perform filling tests to check the differences in filling qualities with the different families obtained and thus identify the ones that make it possible run the process.

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