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## Comparison of two powder conditioning methods to improve $\text{UO}_2$ powder flowability for press die filling

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In the nuclear industry, mixed oxide fuel pellets are manufactured by powder metallurgy process. This work aims at improving the physical and flow properties of nuclear powders in order to obtain a reproducible die filling and produce homogeneous and dense fuel pellets after sintering. The studied powder was a grinded mixture of  $\text{UO}_2$  powder and grinded sintered pellets scraps (recycled). Two powder conditioning methods were tested and compared: a V-blender and an oscillating and rotating OscilloWitt-Lab sieve mill associated to a Turbula mixer. The first conditioning method consisted in three main steps realized in a V-blender: a deagglomeration step with an intensifier bar, a stirring step and a lubrication step. The second conditioning method also consisted in three main steps: a sieving step with an OscilloWitt-Lab, followed by a stirring step and a lubrication step in a Turbula mixer. Different intensifier bar speeds on the V-blender and different sieve mesh sizes on the OscilloWitt-Lab were studied. Particle size, morphology, mechanical properties and flow properties were characterized after each step. The V-blender only slightly improved the flowability compared to the initial powder. The intensifier bar speed had a negligible impact on the physical characteristics and the flowability of the powder. The OscilloWitt-Lab associated to the Turbula mixer allowed a marked improvement of the powder flow properties compared to the initial powder. The sieve mesh size of the OscilloWitt-Lab had a significant impact on the powder physical and flow characteristics. Indeed, using the 250  $\mu\text{m}$  mesh of the OscilloWitt-Lab with stirring and lubrication resulted in the most favorable powder properties. Therefore, it seems to be the most promising conditioning method to obtain reproducible die filling as well as dense and homogeneous fuel pellets.

Keywords: Uranium dioxide powder; Flowability; Powder rheometer; Die filling; Morphological characterisation; Agglomerates uniaxial compression

## 1. Introduction

Nuclear fuels intended for fast-neutron reactors consist of pellets produced by compression of mixed oxide powders (uranium oxide and plutonium oxide). The new generation of fast-neutron reactor fuels will likely have an annular geometry instead of the solid current cylindrical shape. They will also probably have a higher proportion of multi-recycled plutonium (poor fissile isotopy). Under these conditions, the flow properties of the mixed oxide powders must be controlled in order to ensure homogeneous and reproducible sintered pellets. Adequate flow properties would also limit the powder retention, the clogging of pipes. Indeed, this may lead to untimely interruptions in the manufacturing process and an increase in the proportion of rejected pellets. The compaction operation of nuclear powders appears as a key step in the manufacturing process of fuel pellets. This operation is used in various industries and requires several successive steps: feeding from the powder storage hopper, die filling, powder compaction and ejection of the pellets. In order to limit rectification and ensure a good productivity, pellets from a given batch are required to have reproducible mechanical characteristics, weight and dimensions [1]. The density and the mechanical strength of the pellets strongly depend on the filling stage of the process. In addition, good powder flow properties throughout the process is essential to obtain regular shaped pellets. Otherwise, several issues such as bridging in hoppers, fouling of pipes as well as powder consolidation and caking could be encountered [2].

However, the properties of the raw nuclear powder are far from meeting these requirements [3]. In particular, the powder has poor flow properties because of its small particle size. Consequently, a preliminary conditioning step is necessary before proceeding with the compaction operation. The conditioning step must provide the powder with the required properties for an optimal die filling operation. In particular, the obtained powder needs to have good “incipient flow” properties (transition from static to dynamic state) and “dynamic flow” properties. Also, the powder bed properties after filling are important to obtain reproducible and homogeneous pellets (high permeability, homogeneous and compact arrangement of agglomerates in the die press are favorable).

Consequently, the objective of this work was to establish an adequate conditioning method to improve the processing of nuclear powders, **in particular** during the die filling step. More precisely, this work **aimed** to compare the **impact** of two conditioning methods **in improving the flow properties** of a UO<sub>2</sub> powder. Both conditioning methods involved several steps (§2.1), which could modify the powder characteristics such as size, morphology and surface roughness. The physical, mechanical and rheological characteristics were assessed after each **stage of the** process in order to determine the best conditioning method, **ie, conducive to** a reproducible, homogeneous and fast die filling **operation**.

## 2. Materials and methods

**For safety and personal protection when handling UO<sub>2</sub> powders, the conditioning methods and the different powder characterizations were carried out in a glove box, where the Relative Humidity and the Temperature were maintained constant (T = 20 °C and RH = 40%). In addition, a personal dosimetric monitoring was realized for each worker in the laboratory.**

### 2.1. Conditioning methods and equipments

Based on a previous study [3], two conditioning methods were investigated and compared in this work:

- The first conditioning method consisted in three main steps, performed in the V-blender presented in Fig. 1:
  - a deagglomeration step with an intensifier bar,
  - a stirring step (or mixing step),
  - a lubrication step.
- The second conditioning method also included three main steps, performed in an OscilloWitt-Lab associated to a Turbula mixer (Fig. 2):
  - a sieving step with an OscilloWitt-Lab,
  - a stirring step (or mixing step) in a Turbula mixer,
  - a lubrication step in a Turbula mixer.

The V-blender consists of a vessel made of two cylinders with an angle between 75° and 90° [4] (75° in the present study). An intensifier bar can be present along its rotation axis. When the

particles fall into the V-blender during the vessel rotation, they are separated and recombined. The V-blender is mainly used in the pharmaceutical industry [5,6]. This operation generally leads to a modification of the particle morphology, with a limited reduction in particle size [7].

The OscilloWitt-Lab (Frewitt) consists of a rotating blade and a cylindrical sieve. Impacts between the rotor and the particles, as well as between the particles and the screen plate, lead to particle fragmentation [8]. The modification of the physical characteristics of particles depends on the impact intensity and their orientation as they are forced through the sieve. This technology leads to a reduction of the particle sizes and the width of the particle size distribution. It can also modify the particle morphology [8].

The final step, common to both conditioning methods, is the powder lubrication. The objective of this step is to reduce the particle-particle and particle-wall friction during the compaction [9]. The lubricant (zinc stearate) is added to the powder in a very small quantity (0.2 %w). Frictions are minimized through specific structural properties of the lubricant and by the reduction of particle surface roughness [10,11].

## 2.2. Batch preparation

In order to approach future mixed oxide powders while facilitating handling conditions, we used an initial powder made of a grinded mixture of  $\text{UO}_2$  powder and  $\text{UO}_2$  grinded scraps (recycled pellets) in a ball mill during 2 hours (around 59 kg of powder). In this study, the  $\text{PuO}_2$  powder was not used.

The operating conditions of the two powder conditioning methods described in section 2.1 (V-blender with an intensifier bar, and rotating OscilloWitt-Lab sieve associated to a Turbula mixer) are presented in Fig. 3 (which also describes the samples nomenclature). They are divided into three elementary steps: deagglomeration or sieving, stirring and lubrication.

Regarding the first powder conditioning method, the rotational speed of the V-blender vessel was set at 10 rpm during the whole process, whereas the intensifier bar was only used during the first stage for 2 hours in order to deagglomerate the powder. Two operating conditions of the rotational speed of the intensifier bar were tested: 2000 rpm and 4000 rpm. They are respectively noted VB2000

and VB4000 (around 650 g of powder per sample). During the second step, the vessel was in rotation for 15 minutes to ensure powder stirring. Powders resulting from this operation were noted VB2000-S and VB4000-S (around 650 g of powder per sample), depending on the operating conditions of the first step (2000 rpm or 4000 rpm). Finally, 0.2%wt of zinc stearate was added and the vessel was rotated for 10 minutes. Powders were noted VB2000-L and VB4000-L after this final step (around 16 kg of powder per sample). **One advantage of this conditioning method was that all the steps were carried out in the V-blender. Consequently, in an industrial context, no powder transfer would be required, which could avoid common powder handling issues (retention of powder, clogging of pipes, etc.) and thus limit manual operations as well as the radiation dose received by workers.**

Regarding the second powder conditioning method, the rotating OscilloWitt-Lab sieve was used during the first step for sieving. Two operating conditions of the sieve mesh size were compared: 250  $\mu\text{m}$  and 500  $\mu\text{m}$  (around 650 g of powder per sample). Powders obtained after the first stage were noted Frew250 and Frew500, respectively. The subsequent stirring process was carried out in a Turbula mixer at 72 rpm for 15 minutes leading to powders named Frew250-S and Frew500-S (around 650 g of powder per sample). Finally, 0.2%wt of zinc stearate was added and the mixing step was performed in a Turbula mixer at 23 rpm for 10 minutes. The resulted batches were noted Frew250-L and Frew500-L (around 1 kg of powder per sample). **Therefore, this conditioning method required the powder transfer between the OscilloWitt-Lab and the Turbula mixer. In an industrial context, this suggests that the transfer line should be properly designed to avoid the retention of powder or clogging of pipes. It should be noted that oscillating and rotating sieve mills are already used in the nuclear industry with great success and without causing major safety issues.**

## 2.3. Characterization of physical and mechanical properties

### 2.3.1. Particle size distribution and morphology

A laser particle size analyzer (Malvern Mastersizer 3000) with a dry powder disperser (Aero S) was used to determine the particle size distribution of the powder samples. The uncertainty of the measurements was around 10% (based on the standard deviation of several measurements performed on the same sample). Different characteristic diameters were used:  $d_{10}$ ,  $d_{50}$  and  $d_{90}$ . They represent

the particle diameters for which the cumulative volume fraction is equal to 10%, 50% and 90%, respectively. In addition, the Sauter mean diameter ( $d_{3,2}$ ) was calculated and the width of the particle size distribution was evaluated by the Span:

$$Span = \frac{d_{90} - d_{10}}{d_{50}} \quad (1)$$

Three measurements for each sample were performed at a low dispersion pressure (0.1 bar) in order to avoid the destruction of agglomerates.

Powder samples were also observed with an optical microscope (Olympus DSX510) and image analysis were performed using Fiji Is Just Image-J software [12] to determine the elongation. Because of the microscope resolution, only particles with a diameter greater than around 100  $\mu\text{m}$  were considered. Each particle was fitted to an ellipse with the same area as the considered particle and the elongation was calculated as:

$$Elongation = 1 - \frac{\text{minor axis length of the fitted ellipse}}{\text{major axis length of the fitted ellipse}} \quad (2)$$

It should be noted that surface roughness does not affect the elongation value. Image analysis was carried out for around 2000 particles per sample and a cumulative frequency (in number) was recorded.

### 2.3.2. Mechanical properties

Mechanical properties of agglomerates were characterized by uniaxial compression tests, through a compression between two punches in a mechanical testing machine (Instron 3400). An agglomerate was placed on the lower fixed punch and it was crushed by the upper punch at a constant speed of 0.8 mm/min. This experiment allowed to determine the force at breakage  $F$  and to estimate the stress to failure, using the following relation [13]:

$$\sigma_r = \frac{0.7F}{\pi r^2} \quad (3)$$

Thirty agglomerates were analysed for each powder sample. A probabilistic treatment of the data was carried out based on the Weibull theory [14], which allowed the determination of the failure probability of the sample  $P_f$  by the formula:

$$P_f = 1 - \exp \left[ - \left( \frac{\sigma_r}{\sigma_0} \right)^m \right] \quad (4)$$

where  $\sigma_r$  is the stress to failure,  $m$  is the Weibull modulus and  $\sigma_0$  is the Weibull constant.

The Weibull modulus ( $m$ ) is a measure of the variability of defect properties according to samples and is linked to the size of the defects of the agglomerate. Sets with very high Weibull modulus have distinct defects (homogeneous distribution of defects) whereas sets with low Weibull modulus ( $m < 20$ ) have a broader range of defects (heterogeneous distribution of defects). The Weibull constant,  $\sigma_0$ , is a characteristic resistance value of the material [14].

#### 2.4. Characterization of flow properties

Prior to the presentation of techniques for powder flowability assessment, it is worth mentioning that this property remains difficult to characterize. The flowability is not an intrinsic property of a powder because it also depends on the state of the stresses to which the powder is subjected and on the geometry of the handling device. It cannot therefore be defined by a single test or single value [15]. In this regard, two different states of flow must be distinguished [16]. The first state is the “incipient flow” and corresponds to the transition from a static to a dynamic state, *i.e.*, when the powder starts to flow. The second state is the “dynamic flow”, which characterizes the flowability of the powder once it moves. These states are found in all powder transfers (filling and discharge of hoppers or silos, die filling, etc.). In addition, the powder bed properties (permeability and arrangement of agglomerates) are also important.

Moreover, some powder properties are generally more suitable to a good flowability during specific process steps. For example, low flow energy (FT4 powder rheometer), low cohesion and high permeability characterize good flow in hoppers [17]. For gravity die filling, powders must also have high permeability, low cohesion, low compressibility and low friction between the die walls and the powder [1,2,15,17]. In addition, the size and shape of the particles are important to avoid die bridging [2] and segregation [1]. For instance, the incipient failure (corresponding to the transition from a static to a dynamic state) of powders with a large particle size distribution is known to be more difficult [18]. Particle morphology and roughness also affect the balance between interparticle and gravitational forces, and thus the flowability. In fact, spherical and smooth particles have less contact points



between each grain than irregular shape or rough surface particles. This decreases interlocking and friction forces which improves the flowability [18–21]. Indeed, particles with high aspect ratio have higher bulk friction coefficients, the interparticle friction is more important and the flowability is degraded [11,22]. Regarding the manufacture of green pellets, the arrangement of the agglomerates in the die is also important.

In this study, the transition from static to dynamic state (incipient flow) was evaluated by shear tests and the determination of the spontaneous flow diameter. On the other hand, a FT4 powder rheometer test (VFR) was used to evaluate the dynamic flow properties. Finally, the powder bed properties were characterized by the permeability, the Carr index, the Hausner ratio and the compressibility, as described below.

#### 2.4.1. Powder density measurements

Powder density measurements (aerated bulk density and tapped density obtained after 2400 shocks) were carried out with a JEL tapping machine (Stav2003). From these values, the Carr index and the Hausner ratio were determined.

The Carr index  $C$  is defined by [23]:

$$C = \frac{V_0 - V_N}{V_0} \times 100 \quad (5)$$

where  $V_0$  is the initial powder volume and  $V_N$  is the powder volume after 2400 shocks. Only one measurement for each sample was performed because it required around 300 g of powder (and the amount of UO<sub>2</sub> powder available for this study was limited). This index characterizes the compressibility of the powder and is used to predict the powder flowability (Table 1).

In accordance with Geldart's classification, the Hausner ratio  $HR$  is defined by [25]:

$$HR = \frac{\rho_N}{\rho_0} = \frac{V_0}{V_N} \quad (6)$$

where  $\rho_0$  is the aerated bulk density and  $\rho_N$  is the tapped density. This index characterizes the fluidization properties of powders and is also used to characterize the powder flowability (Table 2).

#### 2.4.2. Spontaneous flow diameter

The minimum orifice size for spontaneous flow was determined with the slide device presented in Fig. 4. This device allows the estimation of the powder ability to fill a press die and it characterizes the transition from a static to a dynamic state.

The fuel pellets considered in this work are expected to have an annular geometry, with a central orifice diameter of 2.5 mm. Thus, in order to be representative of the die filling process, an annular geometry was used for the slide device. Six different outlet diameters were considered, as shown in Table 3. The thickness  $e$  corresponds to the space between the central rod (2.5 mm diameter) and the die walls.

In this test, a small cylinder containing the powder is moved along the slide, from the smallest diameter to the largest diameter. The spontaneous flow diameter is defined as the smallest diameter where the powder flows into the orifice. Three measurements were performed for each sample.

#### 2.4.3. FT4 rheometer (Freeman Technology)

The rheological behavior of powders was studied with a FT4 rheometer, through the determination of four properties: permeability, compressibility, shear test and Variable Flow Rate (VFR), which allow a quantitative description of the powder flowability. For each property, three measurements were performed for the initial powder and the final powder after lubrication (VB2000-L, VB4000-L, Frew250-L and Frew500-L), and the corresponding standard deviations were calculated.

##### 2.4.3.1. Permeability

The permeability is the ability of a powder bed to be traversed by a fluid, depending on its porosity and its cohesion. In this study, the permeability was determined using the FT4 powder rheometer. The measurements were carried out in a cylindrical cell with a diameter of 25 mm, a volume of 10 mL and a porous aeration base connected to the aeration module.

Before the measurement, the powder bed was conditioned using the FT4 blade. The blade is then replaced by a porous piston, which applies a constant normal stress of 2 kPa to the powder bed.

Then, an increasing air flow rate, from 0 mm/s to 10 mm/s, was introduced through the powder bed and the pressure drop was measured for each air flow rate. The permeability is defined by:

$$k = \frac{q \mu L}{\Delta P} \quad (7)$$

where  $k$  is the permeability (cm<sup>2</sup>),  $q$  is the air flow rate (cm/s),  $\mu$  is the air viscosity (Pa.s),  $L$  is the length of powder bed (cm) and  $\Delta P$  is the pressure drop across the powder bed (Pa).

#### 2.4.3.2. Compressibility

The compressibility was determined using the FT4 rheometer [24]. The powder bed, placed in a cylindrical cell with a diameter of 25 mm and a volume of 10 mL, was compressed under increasing normal stresses, ranging from 1 kPa to 15 kPa. The volume variation of the powder bed for each normal stress was measured. The compressibility was calculated by the formula:

$$\text{Compressibility} = \frac{\text{Volume variation}}{\text{Initial volume}} \times 100 \quad (8)$$

#### 2.4.3.3. Shear test

Shear tests were realized with the FT4 rheometer [24]. Measurements were performed using a 10 mL cylindrical cell. During the first step (“pre-shearing” step), the powder was compressed under a normal stress  $\sigma_{pre}$  and pre-sheared until it reached a constant shear stress noted  $\tau_{pre}$ . During the second step (shearing step), a normal stress  $\sigma_{sh}$ , which was less than the normal stress  $\sigma_{pre}$ , was applied to the sample. The powder was then sheared and the maximum shear stress  $\tau_{sh}$ , corresponding to the powder bed failure, was evaluated. This second step was realized under different normal stresses  $\sigma_{sh}$  and several couples ( $\sigma_{sh}, \tau_{sh}$ ) were obtained, allowing to plot the yield locus, as presented in Fig. 5-a.

Mohr circles analysis allows different parameters to be determined (Fig. 5):

- The major principal stress  $\sigma_1$  corresponds to the shear stress value of the highest intersection between the larger Mohr circle and the x-axis. It characterizes the maximum stress applied to the powder.
- The unconfined yield strength  $\sigma_c$  corresponds to the shear stress value of the highest intersection between the smaller Mohr circle and the x-axis. It characterizes the normal

stress for which the rupture of the powder bed subjected to uniaxial compression takes place.

- The cohesion  $\tau_c$  corresponds to the y-intercept of the yield locus. It characterizes the cohesion of the powder.
- The flowability index  $ffc$  allows to characterize the flowability and it is determined by :

$$ffc = \frac{\sigma_1}{\sigma_c} \quad (9)$$

By plotting the unconfined yield strength  $\sigma_c$  as a function of the major principal stress  $\sigma_1$  (Fig. 5-b), the flow function is obtained and characterizes the powder flowability [19,26]. In this study, the shear tests were carried out at several pre-consolidation normal stresses ( $\sigma_{pre}$ ): 3 kPa, 6 kPa, 9 kPa and 15 kPa.

#### 2.4.3.4. Variable Flow Rate (VFR)

The Variable Flow Rate test (VFR), realized with the FT4 rheometer [24], characterizes the powder flowability in a dynamic state. Freeman Technology established the following protocol, presented in Fig. 6, which consists of eleven successive tests.

During the seven first tests, the blade had a constant tip speed of 100 mm/s for each measurement cycle. The four last tests were performed at decreasing speeds: 100 mm/s, 70 mm/s, 40 mm/s and 10 mm/s. During each test, the energy required to induce a dynamic flow state was measured. Different coefficients can be defined from these energies (more details can be found in [24]).

The Basic Flow Energy (BFE) is the energy obtained during Test 7. It represents the energy necessary to move a given volume of powder. The Flow Rate Index (FRI) allows the estimation of the impact of the flow speed on the energy and is defined by:

$$FRI = \frac{\text{Energy of test 11 (10 mm/s)}}{\text{Energy of test 8 (100 mm/s)}} \quad (10)$$

### 3. Results and discussion

#### 3.1. Initial powder characteristics

The particle size distribution and the characteristic diameters of the initial powder are presented in Fig. 7 (also Fig. 8) and Table 4, respectively. Table 5 shows the Weibull strength and modulus of the agglomerates found in the initial powder. It should be noted that other results are shown in these Figures and Tables, and will be discussed later in this paper.

The particle size distribution of the initial powder is broad with large agglomerates. These characteristics are not suitable for pellets manufacturing since it would lead to heterogeneous compaction and densification. In fact, the space between the needle and the walls of the press die is 3.3 mm and the agglomerates must have a smaller size. In addition, mechanical properties are an important parameter. The agglomerates must be strong enough to withstand the transfer of powders during the process, but sufficiently fragile to be destroyed during pressing to obtain dense and homogeneous pellets before sintering. The results presented in Table 5 will be discussed later in this paper, when comparing to other powders.

### 3.2. Influence of the V-blender (first conditioning method)

The influence of the three V-blender elementary steps (deagglomeration, stirring and lubrication) on the physical and mechanical characteristics of the powder is studied in relation to the powder flowability. Two intensifier bar speeds are considered: 2000 rpm and 4000 rpm.

#### 3.2.1. *Impact on physical and mechanical properties*

For both intensifier bar speeds, particle size distributions after each elementary step are presented in Fig. 7, characteristic diameters and span are gathered in Table 4. Also, the elongation of agglomerates is reported in Fig. 9. These results show that the deagglomeration step (VB2000 and VB4000) has an effective impact on the particle size and morphology distributions. Compared to the initial powder, after V-blender deagglomeration, the population of particles larger than 500  $\mu\text{m}$  decreases and the main population is around 250  $\mu\text{m}$  for both speed conditions. Furthermore, the speed of the intensifier bar has an important impact on the resulting powder. Indeed, an important increase of the population of fine particles and decrease of the span is observed at 2000 rpm (VB2000), whereas almost no change on fine particles, a higher proportion of millimeter agglomerates and a higher span are observed at 4000 rpm (VB4000). During this deagglomeration step, particles/particles impacts and

particles/wall impacts (between particles and the intensifier bar or the vessel) are generated. These impacts can probably lead to abrasion and fragmentation of particles. In addition, the vessel rotation allows to agglomerate particles. These mechanisms seem to be confirmed by the presence of particles less elongated after this step in both tested conditions. In other words, the initial powder is efficiently deagglomerated and less elongated particles (*i.e.*, more spherical particles) are obtained, particularly at 2000 rpm (VB2000).

During the stirring step (VB2000-S and VB4000-S), an agglomeration of fine particles (corresponding to a lower fine particles proportion and a higher proportion of millimeter agglomerates) is observed whatever the deagglomeration conditions, with a small increase of the span. In fact, the intensifier bar is not used during this step and the rotation of the vessel allows the agglomeration of particles.

Then, the lubrication stage amplifies this phenomenon for VB2000-L whereas it has almost no impact for VB4000-L. The latter result can be explained by the state of VB4000 powder which, as shown previously, was already more agglomerated than VB2000 powder before stirring and lubrication. Thus, the lubrication step seems to have an impact on the particle size depending on the agglomeration state of the powder before lubrication. Moreover, the stirring and the lubrication steps seem to have very little impact on the morphology of the agglomerates in the tested conditions (Fig. 9).

The breaking strengths of agglomerates are reported in Table 5. The Weibull modulus is low ( $m < 20$ ) for both powders (VB2000-L and VB4000-L), which means that the distribution of the defects is heterogeneous and the dispersion of the breaking strengths is high. After deagglomeration, stirring and lubrication with the V-blender, breaking strengths values are similar to those of the initial powder for both deagglomeration conditions (*i.e.*, at 2000 rpm and 4000 rpm). Consequently, the first conditioning method (V-blender) seems to have very little impact on the structure of the agglomerates, which maintain their initial breaking strengths.

Overall, compared to the initial powder, the V-blender conditioning method (VB2000-L and VB4000-L) generates a particle size distribution with a main population around 250  $\mu\text{m}$ , with less fine particles but a higher proportion of millimeter agglomerates. Also, agglomerates are less elongated

(i.e., more spherical) due to the deagglomeration/agglomeration process. However, the structure and mechanical properties of these agglomerates seems similar to the initial powder.

### 3.2.2. Flowability properties

In general, powders have good flow properties when the flow rate index (FRI), the cohesion and the compressibility have low values and when the flowability index  $ffc$  and the permeability are high [15,27]. In addition, a lower Carr index, Hausner ratio [23–25] and spontaneous flow diameter correspond to a better flowability. Finally, it should be noted that the Basic Flow Energy (BFE) is more difficult to interpret [15].

The Carr index, the Hausner ratio and the spontaneous flow diameter are determined after all the steps and are presented in Table 6 (other results are shown and will be discussed later in this paper). The Carr index and the Hausner ratio increase after the deagglomeration step (VB2000 and VB4000). Indeed, powder flow properties are degraded, probably due to a higher proportion of fine particles caused by the intensifier bar. The stirring step (VB2000-S and VB4000-S) and the lubrication step (VB2000-L and VB4000-L) lead to a decrease of the Carr index and the Hausner ratio. Actually, these steps allow particles agglomeration, which reduces the amount of fine particles and as a result improves the flowability. However, the final Carr index and Hausner ratio are not better (i.e., lower) than those of the initial powder. On the other hand, the evolution of the spontaneous flow diameter indicates significant differences between the initial powder and the two final powders after the V-blender conditioning method (VB2000-L and VB4000-L). Indeed, after the deagglomeration step (VB2000 and VB4000), the spontaneous flow diameter slightly increases compared to the initial powder, whereas it decreases after stirring (VB2000-S and VB4000-S) and lubrication (VB2000-L and VB4000-L) for the same reasons as described for the Carr Index and Hausner ratio variations. It is worth noting that the spontaneous flow diameters for the final powders (VB2000-L and VB4000-L) are significantly smaller than that of the initial powder. The more spherical shape of the final powders (Fig. 9), which leads to less friction between particles, could explain this significant improvement (i.e., decrease) of the spontaneous flow diameter.

Measurements with the FT4 powder rheometer were performed on the initial powder and on the two final powders after lubrication (VB2000-L and VB4000-L). Indeed, the available amounts of powders were insufficient to perform these tests after the deagglomeration and stirring steps.

The powder permeability measurements (Fig. 11) show a very small impact of the V-blender conditioning method. Indeed, the VB4000-L powder presents a permeability similar to that of the initial powder and the permeability of the VB2000-L powder is not significantly lower (overlapping standard deviation error bars). In fact, after the V-blender conditioning method, the particle size distributions are wide with coarse and fine particles, similarly to the initial powder (Fig. 7).

The compressibility values at 15 kPa (Fig. 12, samples VB2000-L and VB4000-L) decrease after the V-blender conditioning method for both operating conditions (intensifier bar rotational speed of 2000 rpm and 4000 rpm, respectively). It can be noticed that the VB2000-L powder compressibility is lower than that of VB4000-L powder. This result is probably due to the particles morphology. Indeed, after V-blender conditioning, particles are less elongated (Fig. 9), particularly for the VB2000-L. In addition, the stirring and the lubrication steps probably reduce the surface roughness of agglomerates due to the agglomeration of fine particles. These morphological properties generally improve the initial arrangement of agglomerates by reducing friction between agglomerates. This leads to a more compact initial arrangement of the powder bed, which is then less compressible (lower compressibility).

The flow functions determined from the shear tests are presented in Fig. 13 and the cohesion ( $\tau_c$ ) for each samples is presented in Table 8. The flow function and the cohesion for the VB2000-L powder are similar to that of the initial powder. These results are compliant with the Hausner ratio and the Carr index. For the VB4000-L powder, the flow function and the cohesion are similar to those of the initial powder when low stresses are applied. However, the flow function increases sharply at higher normal stresses, leading to lower  $f f_c$  values when  $\sigma_1$  is higher than around 12 kPa. This result could be explained by a weak agglomerate structure, obtained through the V-blender conditioning method with the intensifier bar speed at 4000 rpm (strong deagglomeration followed by a reagglomeration of the particles). These weak agglomerates might be more prone to abrasion,



increasing the amount of fine particles in the shear region, and thus leading to poorer flow properties (i.e., lower  $ff_c$  and higher cohesion values, shown in Table 8 at  $\sigma_{pre} = 15$  kPa).

The Basic Flow Energy (BFE) and the Flow Rate Index (FRI) are determined from VFR measurements (Fig. 14). It can be observed that the BFE decreases after the V-blender conditioning method (VB2000-L and VB4000-L). This is explained by the stirring step and the addition of lubricant, which make the particles more spherical (Fig. 9). This morphological change decreases the friction between particles. Thereby, the blade needs less energy to move the final powders (VB2000-L and VB4000-L) compared to the initial powder, which explains the decrease of the BFE values. In the same way, the lower FRI values of the final powders (VB2000-L and VB4000-L) are associated with better flow properties, as reported in the literature [24,28].

### 3.2.3. Discussion

To produce green pellets, the first step is the transition from a static to a dynamic state of the powder in order to fill the press die. The shear tests can be considered as representative of this step. With the intensifier bar speed at 2000 rpm (VB2000-L), the flowability index ( $ff_c$ ) and cohesion ( $\tau_c$ ) values are similar to those of the initial powder regardless of the applied stress. With the intensifier bar speed at 4000 rpm (VB4000-L), the flowability index and cohesion values are similar at low stresses but at higher stresses, the flow properties are degraded (i.e., lower  $ff_c$  and higher  $\tau_c$ ) compared to the initial powder. Results obtained from the shear tests seem to show that the V-blender is not appropriate to enhance the incipient flow characteristics (transition from a static to a dynamic state). Nevertheless, the determination of the spontaneous flow diameter (which also estimates the aptitude of the powder to switch from a static to a dynamic state, in order to fill a press die), show that the die filling may be easier after the V-blender conditioning method (VB2000-L and VB4000-L). It should be noted that the powder sample is submitted to much lower stresses during the spontaneous flow diameter tests compared to shear tests. This means that the agglomerates obtained after V-blender conditioning (deagglomeration, followed by a reagglomeration of the particles) might hold their structure during the spontaneous flow diameter tests, while they can be prone to abrasion during shear tests. On the other hand, the spontaneous flow diameter test is probably more representative of the die

filling operation. Therefore, from the results, it can be assumed that the press die filling could be slightly more reproducible and robust after the V-blender conditioning method.

After the transition from a static to a dynamic state, powders are considered to be in dynamic flow until the die is filled. The VFR tests characterize the flow properties of the powder in these conditions. The decrease of the BFE and the FRI show that the energy necessary to move the powder is decreased, so the flowability in a dynamic state is enhanced after the V-blender conditioning method (VB2000-L and VB4000-L), compared to the initial powder. The addition of lubricant and the more spherical shape of the agglomerates, which reduces the friction between particles, can explain this. This flowability improvement **could be** favorable to the reproducibility of the green pellets. The initial intensifier bar rotation speed seems to have no noticeable impact on the dynamic flow properties.

Finally, convenient permeability and arrangement of particles in the press die are important to have an estimation of the powder ability to form homogeneous green pellets. More precisely, a higher permeability is favorable for a homogeneous and reproducible (low volume variation) die filling operation. After V-blender conditioning, the permeability is similar to the initial powder, for both tested conditions (VB2000-L and VB4000-L). In addition, the Carr index and the Hausner ratio are both similar to the initial powder. These results show that, compared to the initial powder, the permeability and the arrangement of particles are not improved after the V-blender conditioning method. However, at 15 kPa, the compressibility decreases, in particular with an intensifier bar speed of 2000 rpm (VB2000-L). Particles are less elongated, so the frictions between particles are lower than the initial powder, which could lead to a better pressing step. Therefore, green pellets **could** be slightly more homogeneous and reproducible after V-blender conditioning.

To conclude, in the tested conditions, the V-blender conditioning method **could** slightly improve the press die filling and **could probably allow the production of** slightly more reproducible and homogeneous pellets.

### 3.3. Influence of the OscilloWitt-Lab associated to the Turbula mixer (second conditioning method)

The three elementary steps of the OscilloWitt-Lab associated to the Turbula mixer are similar to those of the V-blender: sieving, stirring and lubrication. The first step (sieving) is performed in the OscilloWitt-Lab, with a 250  $\mu\text{m}$  sieve mesh or a 500  $\mu\text{m}$  sieve mesh. The stirring and lubrication steps are performed in the Turbula mixer. The influence of these three steps on the physical and mechanical characteristics of the powder is studied in relation to the flowability properties.

### *3.3.1. Impact on physical and mechanical properties*

The sieve mesh size has a strong impact on the particle size distributions (Fig. 8 and Table 4) and particle morphology (Fig. 10). Indeed, with the 250  $\mu\text{m}$  sieve (Frew250), the proportion of fine particles increases whereas the number of particles larger than 500  $\mu\text{m}$  decreases. The main population is around 250  $\mu\text{m}$  and the particles are more elongated compared to the initial powder. With the 500  $\mu\text{m}$  sieve (Frew500), the population of particles larger than 800  $\mu\text{m}$  decreases but the proportion of fine particles and the morphology are not significantly impacted. The main population is logically around 500  $\mu\text{m}$ . In the OscilloWitt-Lab, particles are forced through the sieve, resulting in the formation of smaller and more elongated particles. For a smaller mesh size, the particles are more strongly forced through the sieve. Compared to the initial powder, the particle size distributions are narrower (strong decrease of the Span, as shown in Table 4), in particular with a smaller mesh size (Frew250).

After stirring in a Turbula mixer, the proportion of fine particles decreases and the average particle size increases for the 250  $\mu\text{m}$  sieve (Frew250-S). In these conditions, it can be assumed that this stirring step leads to the agglomeration of fine particles and the lubrication step (Frew250-L) allows a supplementary agglomeration. For the 500  $\mu\text{m}$  sieve, particles size is larger after stirring (Frew500-S) and diminishes to reach 350  $\mu\text{m}$  for the main population after lubrication (Frew500-L). Furthermore, for both sieving conditions, the stirring (Frew250-S and Frew500-S) and lubrication (Frew250-L and Frew500-L) steps performed in the Turbula mixer induce less elongated particles (Fig. 10). This effect seems to be more pronounced with a 250  $\mu\text{m}$  mesh size (Frew250-L).

Powders obtained after the final lubrication step (Frew250-L and Frew500-L) have a significantly higher breaking strength compared to that of the initial powder (Table 5). It can be assumed that particles are consolidated when they are forced through the mesh (sieving step in

OscilloWitt-Lab). As for the V-blender conditioning method, the Weibull modulus is low ( $m < 20$ ) because of a heterogeneous distribution of the defects and a high dispersion of the breaking strengths.

After the “OscilloWitt-Lab + Turbula” conditioning method (Frew250-L and Frew500-L), the millimeter agglomerates are broken, the proportion of fine particles decreases, the main population is around 250  $\mu\text{m}$  and 350  $\mu\text{m}$  respectively and their particle size distributions are narrower (lower span), compared to the initial powder. Particles are less elongated and their breaking strengths are increased.

### 3.3.2. *Flowability properties*

The Carr index, Hausner ratio and spontaneous flow diameter are presented in Table 6. The Carr index and Hausner ratio increase after the sieving step (Frew250 and Frew500), which degrades the powder flowability for both sieving conditions. It can be noted that this impact is greater with the 250  $\mu\text{m}$  sieve (Frew250) than the 500  $\mu\text{m}$  sieve (Frew500). In fact, after the OscilloWitt-Lab sieving step, powders contain more fine particles and are more elongated, which decrease the flowability. The stirring step (Turbula mixer) improves the powder flowability whatever the sieve mesh size used (Frew250-S and Frew500-S). Finally, the lubrication step (Turbula mixer) improves again the flowability for the Frew250-L, whereas it has no significant influence for the Frew500-L. These results are in good agreement with the physical characteristics discussed in the previous section. Indeed, more spherical particles (lower elongation) and a lower span are observed after the stirring and lubrication steps, in particular for the Frew250-L. The evolutions observed with the Carr index and the Hausner ratio are in good agreement with those noted for the spontaneous flow diameter. In fact, the spontaneous flow diameter is larger (= lower flowability) after sieving (Frew250 and Frew500) compared to the initial powder, whereas it is lower after stirring (= better flowability) (Frew250-S and Frew500-S). Finally, the lubrication stage leads to an improvement of the powder flowability for the Frew250-L but has a negligible influence for the Frew500-L.

As for the V-blender experiments, measurements with the FT4 rheometer were carried out on the final powders after lubrication (Frew250-L and Frew500-L). As mentioned before, the available amounts of powders were insufficient to perform these tests after the first two steps (sieving and stirring).

The powder permeability measurements (Fig. 11) show an increase of the permeability for the Frew250-L but no significant impact on the permeability of the Frew500-L powder. Compared to the 500  $\mu\text{m}$  sieving mesh (Frew500-L), the particle size distribution is narrower (much lower Span in Table 4) with the 250  $\mu\text{m}$  sieving mesh (Frew250-L) and the agglomerates are also more spherical (less elongation in Fig. 10). These two properties facilitate the air flow and increases permeability.

The compressibility values at 15 kPa (Fig. 12) indicate a decrease after the “OscilloWitt-Lab + Turbula” conditioning method. The arrangement of agglomerates is initially more homogeneous and compact, which decreases the compressibility. In general, this means that the powder is less cohesive, which is in agreement with the Carr index.

The flow functions determined from the shear tests are presented in Fig. 13. They are lower than that of the initial powder for all normal stresses applied and for both sieving conditions. This corresponds to a significant improvement of the flow properties, particularly when using the 250  $\mu\text{m}$  sieving (Frew250-L). A lower cohesion is also observed for all normal stresses (Table 8). Again, the particle size distributions are narrower (much lower Span in Table 4) after “OscilloWitt-Lab + Turbula” conditioning, and the agglomerates are also more spherical (less elongation in Fig. 10). All these changes decrease the cohesive forces and the friction between particles, leading to an improvement of the flowability properties [18,19,21].

The Basic Flow Energy (BFE) and the Flow Rate Index (FRI) are determined from VFR measurements. The BFE decreases after the OscilloWitt-Lab associated to the Turbula mixer (Frew250-L and Frew500-L). This change can be explained by the presence of less elongated particles and by the action of the lubricant. Both these aspects decrease friction and cohesive forces, which therefore reduce the energy required to move the powder bed (BFE). Similarly, after “OscilloWitt-Lab + Turbula” conditioning, the Flow Rate Index (FRI) is smaller than the initial powder and reach a value close to 1. It has been shown in the literature [24,28] that FRI values close to 1 correspond to powders that are less compressible and less cohesive (better flowability properties).

### 3.3.3. Discussion

As described for the V-blender, different powder flow properties are necessary to manufacture reproducible and homogeneous green pellets: the transition from static to dynamic state, the dynamic

flow, and the powder bed properties after filling (high permeability, homogeneous and compact arrangement of agglomerates in the press die are favorable). Overall, the “OscilloWitt-Lab + Turbula” conditioning method leads to a narrower particle size distribution (lower Span) and more spherical agglomerates, which **should be** favourable characteristics for **the** die filling operation.

The powder aptitude to change from a static to a dynamic state (incipient flow) is characterized by the shear tests and the spontaneous flow diameter. The flow function and the cohesion are lower than those of the initial powder for all stresses applied and for both sieving mesh size tested (Frew250-L and Frew500-L). Furthermore, these results show that the transition from static to dynamic state is easier for the Frew250-L compared to the Frew500-L. After the application of the “OscilloWitt-Lab + Turbula” conditioning method, in both conditions (250  $\mu\text{m}$  and 500  $\mu\text{m}$  mesh), the spontaneous flow diameter is lower (= better flowability) than the value measured for the initial powder. This means that the die filling would **probably** be more efficient with the conditioned powder, in particular with the Frew250-L.

The flowability in a dynamic state is studied through the VFR tests. The BFE and the FRI decrease after the conditioning process (Frew250-L and Frew500-L). Comparing to the initial powder, the energy necessary to move the powder decreases so the flowability is improved. This result implies that the die filling **should** be more reproducible. The initial sieve mesh size seems to have no impact on the dynamic flow.

Finally, the permeability and the homogeneous arrangement of agglomerates in the die are important factors to obtain homogeneous green pellets. The permeability increases for the Frew250-L and is similar to the initial powder for the Frew500-L. A higher permeability is favorable for a homogeneous and reproducible (low volume variation) die filling operation. In addition, the Carr index and the Hausner ratio are lower than the initial powder, in particular for the Frew250-L. Also, at 15 kPa, the compressibility is lower than the initial powder, in particular for the Frew500-L. Thus, the powder has a more homogeneous arrangement in the press die compared to the initial powder, which **could lead to** more reproducible and homogeneous green pellets.

To conclude, in the tested conditions, the “OscilloWitt-Lab + Turbula” conditioning method clearly improves all the characteristics required for a performant die filling operation, which **should**

normally lead to reproducible and homogeneous pellets. This improvement is particularly pronounced with the 250  $\mu\text{m}$  sieving mesh of the OscilloWitt-Lab (Frew250-L).

#### 3.4. V-blender or OscilloWitt-Lab associated to the Turbula mixer: what is the best compromise?

The objective is to produce reproducible, homogenous and dense green pellets. For that, final powders, after the lubrication stage, are compared in this work. Table 9 summarizes the powder flow properties depending on the steps necessary to produce green pellets.

In the tested conditions, for the V-blender and “OscilloWitt-Lab + Turbula” conditioning methods, the powder flowability is globally improved compared to that of the initial powder. The “OscilloWitt-Lab + Turbula” conditioning (Frew250-L and Frew500-L) allows a pronounced improvement, whereas the V-blender (VB2000-L and VB4000-L) induces slight modifications with limited impact on the flow properties.

The transition from static to dynamic state (incipient flow), the dynamic flow and the homogeneous arrangement of agglomerates are both important to produce reproducible, homogeneous and dense green pellets. A compromise between all these properties is necessary. In the tested conditions, the OscilloWitt-Lab with a 250  $\mu\text{m}$  sieve, associated to stirring and lubrication in the Turbula mixer (Frew250-L), is clearly the best conditioning method to improve the flow properties (Table 9). **However, it should be noted that in an industrial context, this conditioning method would require the powder transfer between the sieve mill and the mixer.**

## 4. Conclusions and perspectives

The objective of this work was to study the influence of two conditioning methods, each **including three** main steps, to improve the flowability of a  $\text{UO}_2$  powder. The physical, mechanical and rheological characteristics were considered after each step in order to determine the best conditioning method to obtain reproducible die filling and homogeneous fuel pellets.

The V-blender modified the particle size distribution and agglomerates were less elongated. The flowability in a dynamic state and the compressibility were only slightly improved. However, the intensifier bar speed had a negligible impact on the physical characteristics and flowability. **One**

advantage of this conditioning method was that all the steps were carried out in the V-blender, which means that no powder transfer was required.

The “OscilloWitt-Lab + Turbula” conditioning method significantly modified the powder characteristics. The particle size distributions were narrower (much lower Span), the main population was shifted towards the OscilloWitt-Lab. After all three steps (sieving, stirring and lubrication), powders were more spherical (less elongated), had better mechanical strengths and were less cohesive, which means that the powder flowability was enhanced. The initial sieve mesh size had a noticeable impact on the particle size distribution and the elongation of the agglomerates.

After “OscilloWitt-Lab + Turbula” conditioning with a 250  $\mu\text{m}$  mesh, a clear improvement of the flowability was observed compared to the other powders. These physical characteristics and flow properties could probably be suitable to ensure a reproducible die filling and to obtain a dense and homogeneous pellet microstructure after sintering. In an industrial context, this conditioning method would require the powder transfer between the sieve mill and the mixer. However, oscillating and rotating sieve mills are already used in the nuclear industry with great success and without causing major safety issues. Overall, these processing conditions seemed to be the best compromise for fuel pellets production.

In order to verify the efficiency of the conditioning method, additional experiments are being carried out to study the reproducibility of the filling of the press die and the densification of the granules after sintering. In addition, the mechanical characterizations did not seem sufficient to explain the behavior of the powder. Additional tests are being carried out to characterize the abrasion properties of the powder. All these complementary characterizations would allow to better anticipate the transposition to real powders, which could have different characteristics and properties owing to their  $\text{PuO}_2$  content.



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(a)



(b)

*Fig. 1: Fist conditioning method – Pictures of the V-blender (a) and the intensifier bar located along its rotation axis (b)*



(a)



(b)

*Fig. 2: Second conditioning method – Pictures of the OscilloWitt-Lab (a) and of the Turbula mixer (b)*

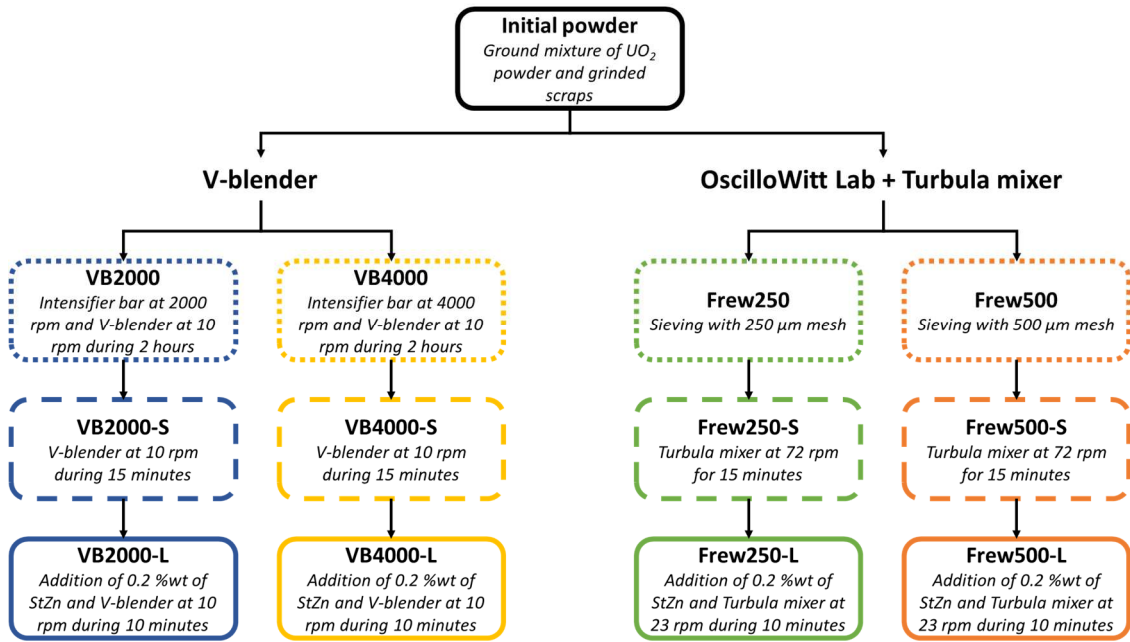
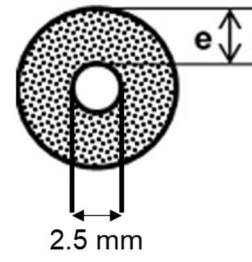


Fig. 3: Definition of the two powder conditioning methods and samples nomenclature



*Fig. 4: Slide for spontaneous flow diameter measurements*

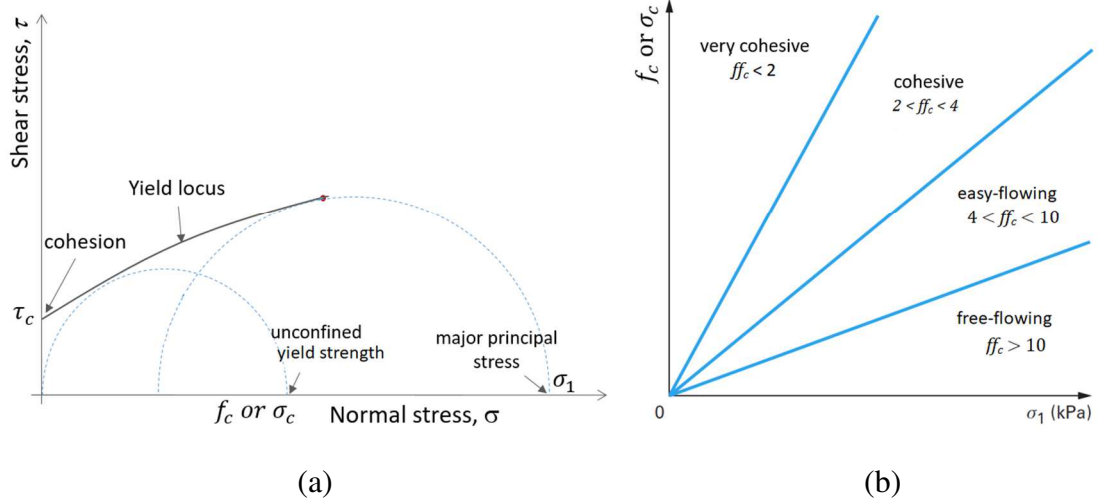
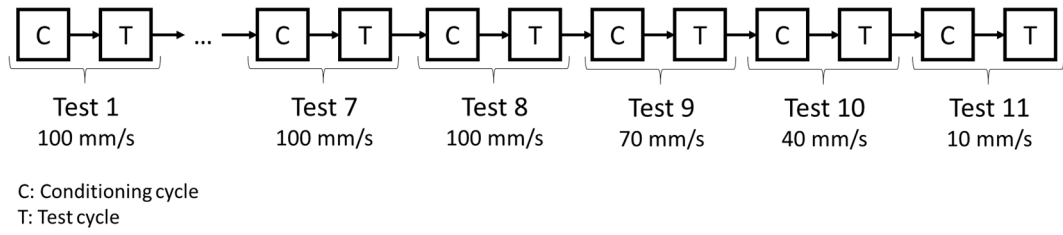


Fig. 5: The shear test. (a) yield locus and Mohr circles analysis, (b) flow function and Jenike classification [19]





*Fig. 6: Protocol of the VFR test*

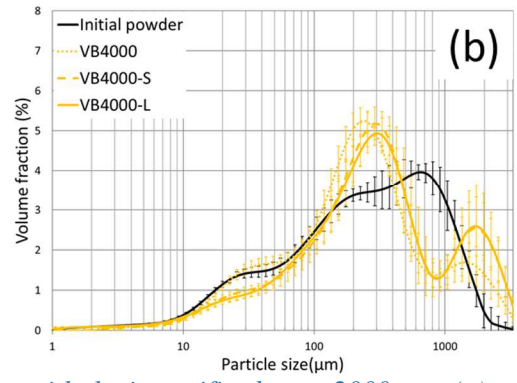
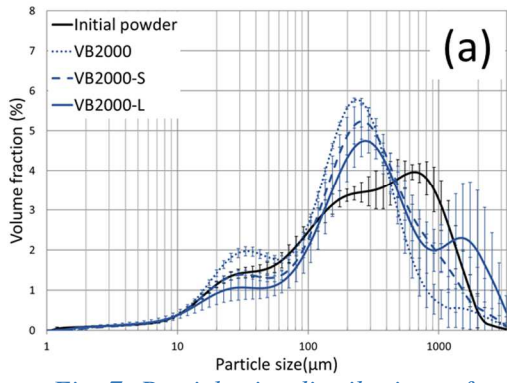


Fig. 7: Particle size distributions after V-blender with the intensifier bar at 2000 rpm (a) and 4000 rpm (b)

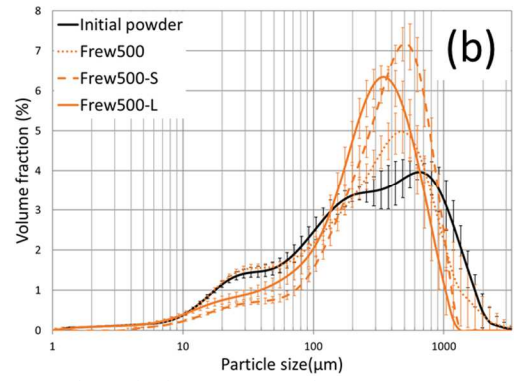
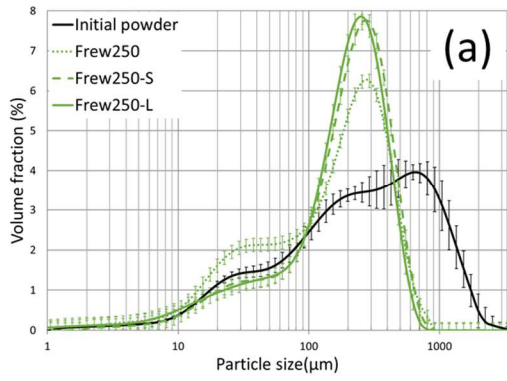


Fig. 8: Particle size distributions after OscilloWitt-Lab with the sieve mesh size of 250  $\mu\text{m}$  (a) and 500  $\mu\text{m}$  (b) and the Turbula

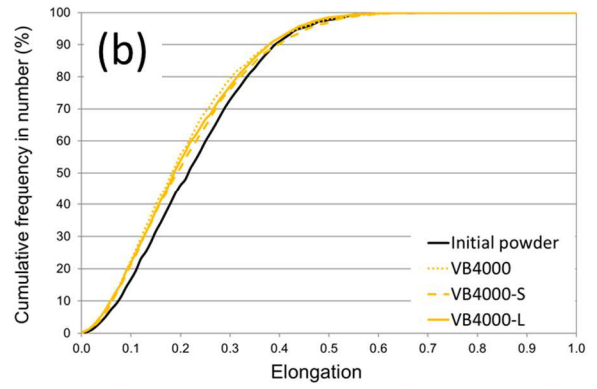
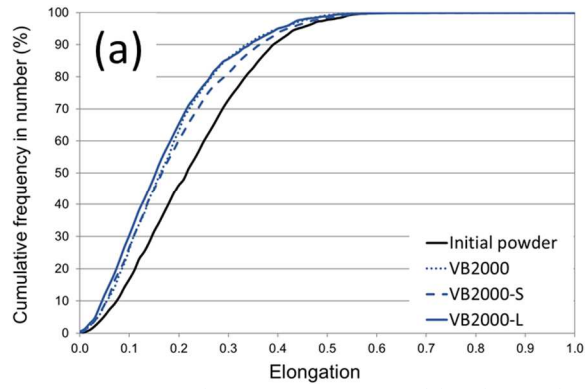


Fig. 9: Elongations after V-blender with the intensifier bar at 2000 rpm (a) and 4000 rpm (b)

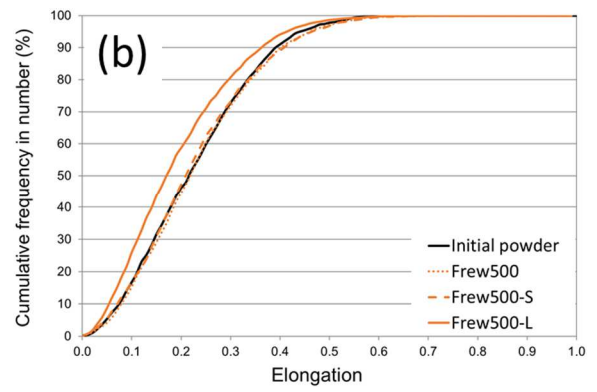
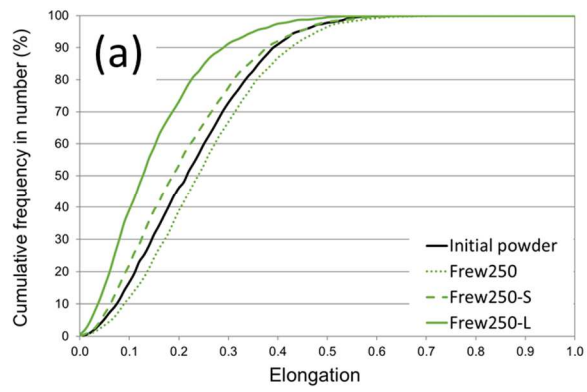
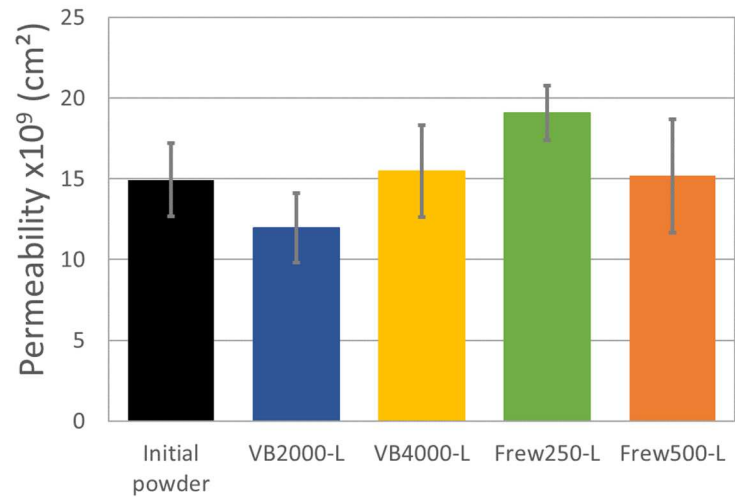
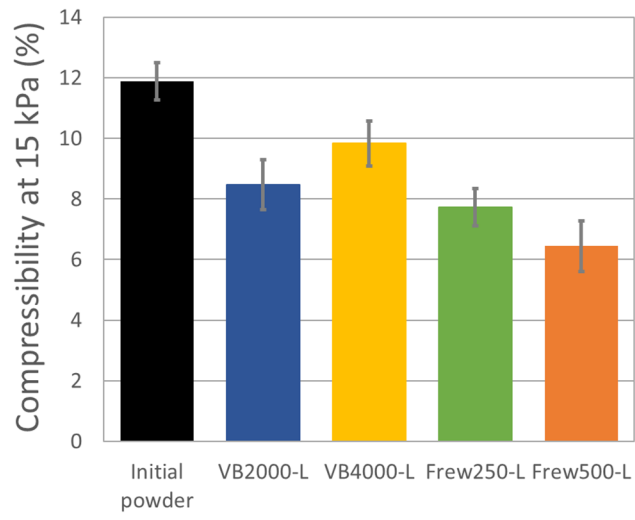


Fig. 10: Elongations after OscilloWitt-Lab with the sieve mesh size of 250  $\mu\text{m}$  (a) and 500  $\mu\text{m}$  (b)



*Fig. 11: Permeability measured with the FT4 rheometer*



*Fig. 12: Compressibility at 15 kPa measured with the FT4 rheometer*

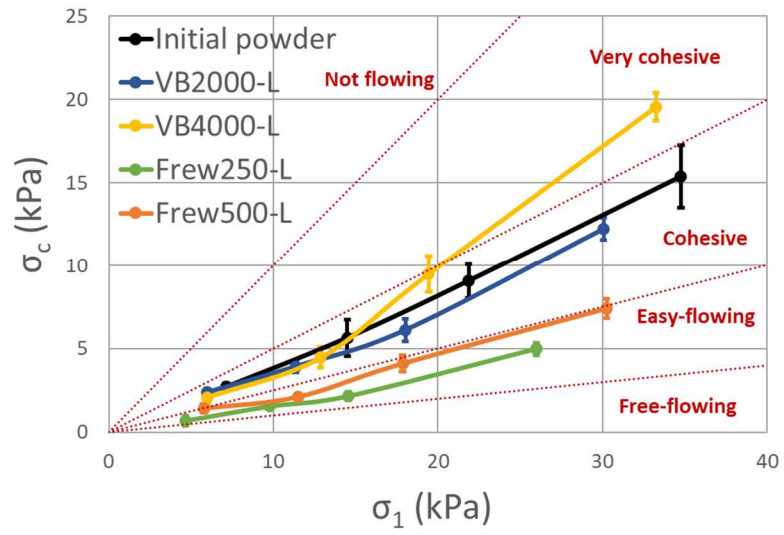
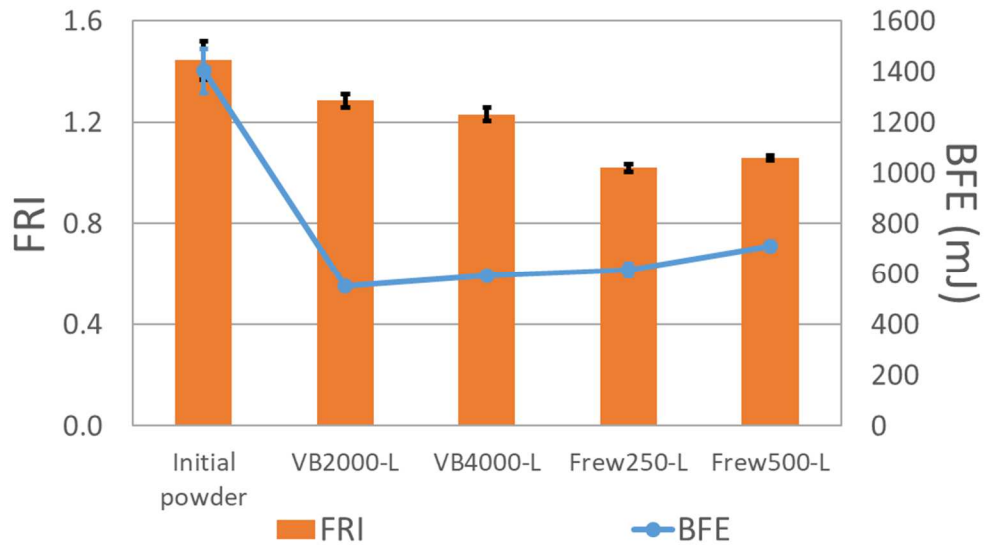


Fig. 13: Flow functions obtained by shear tests





*Fig. 14: VFR results measured with the FT4 rheometer*

*Table 1: Carr index's classification [25]*

Carr index $C$	Powder flowability
$5 < C < 15$	Good flow
$15 < C < 18$	Fair flow
$18 < C < 22$	Passable flow
$22 < C < 35$	Poor flow
$35 < C < 40$	Very poor flow

*Table 2: Hausner Ratio's classification [25]*

Hausner Ratio $HR$	Powder flowability
$HR > 1.4$	Powder classified in group C : cohesive powders
$HR < 1.25$	Powder classified in group A : non-cohesive powders
$1.25 < HR < 1.4$	Intermediate behavior

*Table 3: Dimensions of the slide*

Number of the orifice	1	2	3	4	5	6
Diameter (mm)	6.05	7.66	9.07	10.59	12.10	15.10
Diameter of the rod (mm)	2.50					
e (mm)	1.78	2.58	3.29	4.05	4.80	6.30

Table 4: Characteristic diameters and span obtained by laser granulometry

Sample	$d_{10}$ ( $\mu\text{m}$ )	$d_{50}$ ( $\mu\text{m}$ )	$d_{90}$ ( $\mu\text{m}$ )	$d_{3,2}$ ( $\mu\text{m}$ )	Span
Initial powder	$28 \pm 2$	$259 \pm 41$	$1021 \pm 143$	$61 \pm 6$	$3.86 \pm 0.41$
VB2000	$26 \pm 2$	$185 \pm 8$	$567 \pm 75$	$62 \pm 6$	$2.92 \pm 0.27$
VB2000-S	$29 \pm 2$	$235 \pm 26$	$932 \pm 397$	$70 \pm 6$	$3.76 \pm 1.20$
VB2000-L	$36 \pm 14$	$282 \pm 69$	$1423 \pm 620$	$72 \pm 19$	$4.79 \pm 1.55$
VB4000	$32 \pm 3$	$217 \pm 28$	$1173 \pm 295$	$77 \pm 7$	$5.22 \pm 0.80$
VB4000-S	$45 \pm 11$	$283 \pm 61$	$1597 \pm 385$	$99 \pm 22$	$5.47 \pm 0.77$
VB4000-L	$39 \pm 7$	$275 \pm 42$	$1653 \pm 246$	$58 \pm 22$	$5.94 \pm 1.01$
Frew250	$22 \pm 1$	$168 \pm 19$	$416 \pm 21$	$44 \pm 4$	$2.36 \pm 0.16$
Frew250-S	$33 \pm 2$	$207 \pm 10$	$428 \pm 10$	$74 \pm 3$	$1.90 \pm 0.05$
Frew250-L	$27 \pm 1$	$194 \pm 7$	$396 \pm 18$	$47 \pm 1$	$1.91 \pm 0.03$
Frew500	$28 \pm 1$	$269 \pm 23$	$861 \pm 161$	$63 \pm 2$	$3.09 \pm 0.40$
Frew500-S	$70 \pm 10$	$358 \pm 33$	$772 \pm 38$	$130 \pm 13$	$1.97 \pm 0.11$
Frew500-L	$38 \pm 8$	$266 \pm 36$	$640 \pm 68$	$67 \pm 13$	$2.27 \pm 0.10$

*Table 5: Weibull strength and modulus of agglomerates*

	<b>Initial powder</b>	<b>VB2000-L</b>	<b>VB4000-L</b>	<b>Frew250-L</b>	<b>Frew500-L</b>
<b>Weibull strength <math>\sigma_0</math> (kPa)</b>	139	102	122	268	320
<b>Weibull modulus m</b>	3.6	4.7	3.4	4.8	3.9

Table 6: Results of the Carr index, the Hausner ratio and the spontaneous flow diameter

Sample	Carr Index	Hausner Ratio	Spontaneous flow diameter (mm)
Initial powder	$20 \pm 2$	$1.26 \pm 0.3$	9.07
VB2000	$30 \pm 2$	$1.44 \pm 0.3$	10.59
VB2000-S	$24 \pm 2$	$1.32 \pm 0.3$	9.07
VB2000-L	$19 \pm 2$	$1.24 \pm 0.3$	6.05
VB4000	$31 \pm 2$	$1.45 \pm 0.3$	9.07
VB4000-S	$25 \pm 2$	$1.34 \pm 0.3$	7.66
VB4000-L	$22 \pm 2$	$1.28 \pm 0.3$	6.05
Frew250	$43 \pm 2$	$1.76 \pm 0.5$	12.1
Frew250-S	$19 \pm 2$	$1.23 \pm 0.3$	7.66
Frew250-L	$14 \pm 2$	$1.17 \pm 0.2$	6.05
Frew500	$27 \pm 2$	$1.37 \pm 0.3$	12.1
Frew500-S	$18 \pm 2$	$1.22 \pm 0.3$	7.66
Frew500-L	$18 \pm 2$	$1.23 \pm 0.3$	7.66

*Table 7: Bulk density before and after compression at 15 kPa*

	<b>Initial powder</b>	<b>VB2000-L</b>	<b>VB4000-L</b>	<b>Frew250-L</b>	<b>Frew500-L</b>
<b>Bulk density before compression (g/mL)</b>	3.20 ± 0.04	3.14 ± 0.05	2.97 ± 0.09	3.49 ± 0.03	3.62 ± 0.04
<b>Bulk density after compression (g/mL)</b>	3.58 ± 0.03	3.45 ± 0.06	3.26 ± 0.08	3.76 ± 0.04	3.86 ± 0.07



*Table 8: Cohesion and flowability index at  $\sigma_{pre}$  values of 3 kPa and 15 kPa obtained by shear tests*

	Initial powder	VB2000-L	VB4000-L	Frew250-L	Frew500-L
Cohesion at 3 kPa	0.63 ± 0.06	0.72 ± 0.08	0.59 ± 0.11	0.22 ± 0.10	0.47 ± 0.13
Cohesion at 15 kPa	3.68 ± 0.52	3.21 ± 0.24	5.48 ± 0.29	1.47 ± 0.11	1.95 ± 0.12
$ff_c$ at 3 kPa	2.64 ± 0.13	2.52 ± 0.01	2.90 ± 0.43	5.61 ± 0.68	4.14 ± 0.73
$ff_c$ at 15 kPa	2.28 ± 0.20	2.47 ± 0.13	1.70 ± 0.12	5.24 ± 0.37	4.10 ± 0.21

Table 9: Comparison of the powder flowability



<b>Transition from static to dynamic state</b>	Shear tests	Low stress	VB2000-L	Initial powder / VB4000-L		Frew500-L	Frew250-L
		High stress	VB4000-L	Initial powder	VB2000-L	Frew500-L	Frew250-L
	Spontaneous flow diameter		Initial powder	Frew500-L	VB2000-L / VB4000-L / Frew250-L		
<b>Dynamic flow</b>	BFE		Initial powder	Frew500-L	VB4000-L	Frew250-L	VB2000-L
	FRI		Initial powder	VB2000-L	VB4000-L	Frew500-L	Frew250-L
<b>Powder bed properties after filling</b>	Permeability		VB2000-L	Initial powder	VB4000-L	Frew500-L	Frew250-L
	Carr index		VB4000-L	Initial powder	VB2000-L	Frew500-L	Frew250-L
	Hausner ratio		VB4000-L	Initial powder	VB2000-L	Frew500-L	Frew250-L
	Compressibility		Initial powder	VB4000-L	VB2000-L	Frew250-L	Frew500-L

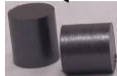


Ball  
milling

**Powder  
conditioning  
method**

Compaction

Sintering



**V-blender**



**OscilloWitt-Lab + Turbula**



**Physical  
characteristics:**

- *Particle size distribution*
- *Morphology*

**Mechanical  
characteristics:**

- *Stress to failure*

**Flow properties:**

- *Carr Index and Hausner Ratio*
- *Spontaneous flow diameter*
- *Permeability*
- *Compressibility*
- *Shear tests*
- *Variable Flow Rate*

**Best compromise :**

**OscilloWitt-Lab + Turbula  
with a 250  $\mu\text{m}$  sieving mesh**

- *Narrower particle size distributions and more spherical agglomerates*
- *Higher mechanical strength*
- *Better flowability*