Measuring the flowing properties of powders and grains

G. Lumay a,b,d,⁎, F. Boschini a,c, K. Traina a,c, S. Bontempi a, J.-C. Remy b, R. Cloots a,c, N. Vandewalle a,b

a APTIS, Institute of Physics, University of Liege, Sart-Tilman, B-4000 Liège, Belgium
b GRASP, Institute of Physics, University of Liege, B-4000 Liège, Belgium
c LCSGreenMat, Institute of Chemistry, University of Liege, B-4000 Liège, Belgium
d F.R.S.-FNRS, B-1000 Bruxelles, Belgium

Abstract

A granular material is a complex system which exhibits non-trivial transitions between the static, the quasi-static and the dynamical states. Indeed, an assembly of grains can behave like a solid or a fluid according to the applied stress. In between solid and fluid granular states, very slow dynamics are observed. When a complete macroscopic characterization of a powder is needed, all these granular states have to be precisely analyzed. In this paper, we show how three measurement techniques can be used to measure the physical properties of a powder. The measurements are based on classical tests modified to meet the recent fundamental researches on granular materials. The static properties of the powder are analyzed through the shape of a heap. The quasi-static behavior is studied with the analysis of the compaction dynamics. Finally, the dynamical regime is monitored through the flow in a rotating drum. In order to illustrate how these measurements can be used in practical cases, analyses are performed with three types of granular materials: silicon carbide abrasives, flours and rice. These selected materials allow to show the influence of the different parameters (gran size, grain size distribution, grain shape) on the macroscopic properties of the assembly. Moreover, these studies show the pertinence of the parameters obtained with the proposed techniques for the rheological characterization of powders and grains.

© 2012 Elsevier B.V. All rights reserved.

1. Introduction

Granular materials are ubiquitous in our daily lives and in industrial applications. Indeed, almost every consumer product has been in a granular state during its manufacturing process. However, in comparison with liquids and gasses, our understanding of the granular state properties is poor. Therefore, any progress in the understanding of the granular material behaviors could have huge consequences for the industry. In particular, the measurement of the physical properties of granular materials is an essential step during the development or the optimization of an industrial process. In addition, the scaling between the results obtained from the measurements in the laboratory and the actual behavior of the powder in the production process represents a difficult transition.

The annual number of scientific publications dedicated to granular materials has increased from 30 in 1990 to 480 in 2010 [1]. A significant part of these publications are related to fundamental studies which are either theoretical or experimental [2–4]. During the last decade, granular material has aroused strong interest in the physics community due to the links with glassy systems and due to puzzling behaviors like phase segregation [5], jamming transition [6,7], complex rheological properties [8], etc. On the other hand, some publications are strongly oriented to the applications. According to the authors in this study, the link between the fundamental studies and the applied studies is often too weak. For instance, when physicists are studying fundamentally the phase segregation process [5], engineers are involved in the improvement of the mixing process [9] and the cross references between respective publications are poor. A long time has passed between the fundamental study of a process involving granular material and the valorization of the fundamental knowledge in applications. In particular, the methods used to characterize the physical properties of granular materials can be strongly improved considering the recent fundamental research. This point will be discussed in details.

A granular material is a complex system because its behavior depends on the applied stress. When a collection of grains forms a pile in a container, the material can be roughly compared to a solid. This is the static state. However, if the container is sufficiently tilted, the granular material starts to flow [10]. In this dynamic state, the layer of grains located at the surface could be seen as a liquid. Between the static and the dynamic states, a granular assembly can experience a quasi-static state. Indeed, if the pile is gently vibrated, the grains rearrange themselves inside the packing and the volume of the pile decreases slowly. This slow dynamics has been the subject of many studies in the physics community [12–18]. Finally, if the container is strongly agitated, the grains experience free flights between some collisions with other grains and with the container walls. In this case, the assembly behaves like a
dissipative gas. In any process, the granular materials can experience some complex transitions between the static, the quasi-static and the dynamic states.

The motion and the jamming of the grains inside a pile are influenced by (i) the steric repulsions, (ii) the friction forces and (iii) the cohesive forces. The interactions with the surrounding fluid also influence the behavior of the grains inside the packing but this effect will not be discussed further in this paper. The steric repulsion is related to the geometry of the grains. The friction forces are influenced by the surface properties and by the chemical nature of the grains. Finally, the cohesive forces are induced by the presence of liquid bridges [19–21], electric charges [22], van der Waals interactions [23] and magnetic dipole–dipole interactions [24–28]. The granular materials can be split in two categories: cohesive and non-cohesive granular materials. In non-cohesive granular materials, the interaction between the grains is mainly related to the steric repulsion and to the friction forces. Therefore, the macroscopic properties of the assembly are governed by the geometry of the grains (shape and size distribution) and by the surface properties of the grains. When the cohesive forces between the grains become greater than the weight of one grain, the macroscopic properties are strongly influenced by the cohesion inside the packing [11]. Therefore, when the grains become smaller, the macroscopic properties of the assembly are mainly governed by the cohesive forces.

Due to the complexity of granular materials, any single and simple test method cannot characterize completely the physical properties of a powder [29,30]. Indeed, the measurement method to analyze the static, quasi-static and dynamic properties of a powder is strongly different. Six methods are commonly reported to measure the powder flowability: the angle of repose [31,32], compressibility index (or Hausner ratio) [33], flow in a rotating drum [34,35], flow through an orifice [37], shear cell [38] and powder rheometers [39]. In addition, many variations of these methods are available. A measuring method has to

### Table 1

<table>
<thead>
<tr>
<th>Flow property</th>
<th>Angle of repose (°)</th>
<th>Hausner ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Excellent</td>
<td>25–30</td>
<td>1.00–1.11</td>
</tr>
<tr>
<td>Good</td>
<td>31–35</td>
<td>1.12–1.18</td>
</tr>
<tr>
<td>Fair</td>
<td>36–40</td>
<td>1.19–1.25</td>
</tr>
<tr>
<td>Passable</td>
<td>41–45</td>
<td>1.26–1.34</td>
</tr>
<tr>
<td>Poor</td>
<td>46–55</td>
<td>1.35–1.45</td>
</tr>
<tr>
<td>Very poor</td>
<td>56–65</td>
<td>1.46–1.59</td>
</tr>
<tr>
<td>Very very poor</td>
<td>&gt;66</td>
<td>&gt;1.60</td>
</tr>
</tbody>
</table>

### Fig. 1

Organization of the paper. After an introduction on the physics of granular materials, methods of powder characterization based on 3 measurement techniques (GranuHeap, GranuPaq and GranuDrum) are proposed. This approach based on the association of three techniques allows to investigate different granular states: the static, the quasi-static and the dynamical states. More details about the measurement techniques are available on the APTIS website [41]. The analysis of three common granular materials shows the pertinence of the parameters obtained with the proposed techniques for the rheological characterization of powders and grains.

### Fig. 2

Two typical heap shapes. (a) Conical heap shape obtained with a non cohesive granular sugar. (b) Irregular heap of powdered sugar which is a cohesive granular material.

### Fig. 3

Illustration of the GranuHeap measurement. The heap is formed on a cylindrical support. A CCD camera takes pictures of the pile for different orientations. Then, a dedicated algorithm finds the position of the interface powder/air by image analysis. Finally, the repose angle $\alpha_r$ and the deviation $\sigma_r$ from an ideal triangular heap (dashed lines) shape are computed.
be easy to use, reproducible and sensitive. Furthermore, the results obtained from the measurements have to be meaningful, easily interpretable and easily usable to improve an industrial process. Table 1 shows the supposed relations between both angle of repose and Hausner ratio and the flowability of the powder. These two simple tests and these fully empirical relations are commonly used to have an idea about the macroscopic properties of a powder. However, this is not sufficient if a precise physical characterization of the powder is needed.

In this paper, we show how three revisited techniques can be used to characterize the macroscopic properties of granular materials (see Fig. 1). The first method (GranuHeap), based on the classical repose angle measurement, gives information about the static properties of the assembly. The second one (GranuPaq), based on the well known tapped density measurement, estimates the quasistatic properties of a granular pile. Finally, the GranuDrum quantifies the flow properties of a granular material. To illustrate how these measurements can be used in practical cases, three types of granular materials have been analyzed. These granular materials are commonly encountered and easy to find in the market: silicon carbide abrasives, flours and rice. These selected materials allow to show respectively the influence of the grain size, grain size distribution and grain shape on the macroscopic properties of the assembly. Moreover, our studies show the pertinence of the parameters obtained with the proposed techniques for the rheological characterization of powders and grains.

Fig. 4. Sketch of the GranuPaQ measurement. The powder is placed in a metallic tube with an initialization protocol described in the text. A light hollow cylinder is placed on the top of the pile to keep it flat during the compaction process. The taps correspond to successive free falls of the tube over a distance $Z$. After each tap, the height $h$ of the pile is measured. Finally, a compaction curve is obtained.

When grains are poured on a surface, they form a heap. Fig. 2 shows two heaps of sugar. While the granular sugar forms a classical conical heap, the powdered sugar forms a strongly irregular heap. Contrary to the granular sugar, the powdered sugar is strongly cohesive due to the small size of the grains. This example clearly shows that the heap shape strongly depends on the grain properties. In particular, a cohesive granular material gives a high value of the repose angle and strong deviations from the conical shape [42]. Therefore, a precise measurement of the heap shape gives some useful information about interactions between the grains.

The angle of repose test is very sensitive to the method used to create the heap. Therefore, an initialization protocol has to be defined. Moreover, after the heap formation, the measurement of the repose angle is not obvious. As shown in Fig. 2, the shape of the heap has to be analyzed carefully. Therefore, the classical method which consists in measuring the heap height $h$ on a circular support of diameter $b$ and in calculating the angle with the relation $\tan(\alpha) = 2h/b$ is subject to caution.

In our measurements, an initialization tube with an internal diameter equal to the support diameter is placed on the support. After filing manually the tube with the sample of powder, the initialization tube goes up at a constant speed of 5 mm/s. Therefore the powder flows from the tube to form a heap on the cylindrical support (see Fig. 3). This support can rotate slowly around its axis. Then, a CCD camera is able to take pictures of the heap for different orientations. To obtain the results presented in the next sections, 8 images separated by a rotation of 22.5° were recorded. In this way, even if the heap shape is complex and asymmetric, we are able to extract all the geometrical information. From each picture of the heap, a dedicated algorithm finds the position of the interface powder/air by image analysis. The repose angle $\alpha$ is the angle of the isosceles triangle which has the same surface area as the heap. The isosceles triangle corresponds to the ideal heap shape. In addition to this parameter, a cohesive index

Fig. 5. Two typical compaction curves presenting the evolution of the packing fraction $\eta$ as a function of the tap number $n$. In this particular case, the initial $\eta_0$ and the final $\eta_{1500}$ packing fractions are the same for both compaction curves. Therefore, the Hausner ratios are identical. The only difference between the two curves is the compaction characteristic time $\tau$ (see Eq. (1)).

2. Measurement methods

2.1. Heap shape measurement

Fig. 6. Two typical flows in a rotating drum. (a) Regular flow obtained with a non cohesive granular sugar. (b) Irregular flow of powdered sugar which is a cohesive granular material.

Fig. 7. Sketch of the GranuDrum measurement. A cylinder is half-filled with the powder and rotates around his axis. For each rotating speed, a CCD camera takes 50 successive images of the flow. A dedicated algorithm enables to measure the flowing angle $\alpha_f$ and the fluctuations $\sigma_f$ of the flow from the shape of the granular/air interface.
\( \sigma \) is measured. This index \( \sigma \) corresponds to the deviation between the heap interface and the ideal heap shape. For a non-cohesive powder, \( \sigma \) is close to zero.

2.2. Compaction measurement

The Hausner ratio measurement is very popular for powder characterization because of its simplicity and its rapidity. The recommended procedure to measure the Hausner ratio is the following [40]. A powder sample of 100 g is gently poured in a 250 ml glass cylinder. Then, the initial volume \( V_0 \) of the powder is measured by naked eyes. Afterward, the pile experiences 500 calibrated taps and the final tapped volume \( V_f \) is measured. The Hausner ratio is calculated with the relation \( HR = V_0 / V_f \). Table 1 shows the empirical relations between the obtained Hausner ratio and the flowability of the powder. This simple test has three major drawbacks. First, the result of the measurement depends on the operator. Indeed, the filling method influences the initial volume \( V_0 \). Second, the volume measurements by naked eyes induce strong errors on the results. Finally, with this simple method, we completely miss the compaction dynamics between the initial and the final measurements.

We have developed a protocol and an automated device (GranuPaq) to perform an accurate measurement of the behavior of a granular material submitted to a series of taps. The measurement is done in a metallic tube to avoid the accumulation of electric charges during the measurement. For that, all the parts of the device are connected to the earth. The diameter and the length of the tube used to perform the studies presented in this paper are \( D = 26 \) mm and \( L = 100 \) mm for the analysis of the abrasives and of the flours and \( D = 35 \) mm and \( L = 150 \) mm for the analysis of the rice. In order to obtain a reproducible and spatially homogeneous initial packing, the following initialization protocol is used. A narrower and bottomless tube is inserted into the measurement tube. Afterward, the initialization tube is filled with the granular material and is removed upward at a low and constant velocity \( v = 1 \) mm/s leaving the grains to rearrange themselves in the measurement tube. Then a light (11.5 g) aluminum hollow cylinder is gently placed on the top of the pile to keep it flat during the compaction process (see Fig. 4). To apply a tap on the packing, the tube goes up to a height of \( Z = 3 \) mm and experiences a free fall over the same height. After each tap, a distance sensor measures the position of the hollow cylinder. From this distance, the height \( h \) and the volume \( V \) of the pile are computed. As the introduced mass of powder is known, the bulk density \( \rho_{bulk} \) evolution as a function of the tap number \( n \) is calculated. The bulk density is the ratio between the mass \( m \) and the volume \( V \) of the powder. The packing fraction \( \eta \) is also calculated by dividing the bulk density \( \rho_{bulk} \) by the true density \( \rho_{true} \) of the material. The fraction \( \eta \) corresponds to the ratio between the volume occupied by the grains and the volume of the pile.

Fig. 5 shows a sketch of two typical compaction curves. The Hausner ratio extracted from both curves has exactly the same value. However, the compaction dynamics are clearly different. Therefore, the Hausner ratio is not sufficient to characterize the powder. An additional parameter is needed. We propose to measure a characteristic number of taps \( n_{1/2} \) needed to reach the middle of the compaction process, i.e. to reach \( \eta = (\eta_{500} + \eta_1) / 2 \). The second method is based on the fit with a physical model [12,18]:

\[
\eta(n) = \eta(\infty) - \frac{\eta(\infty) - \eta(0)}{1 + \ln(1 + \frac{n}{\tau})} \tag{1}
\]

where the fitting parameters \( \eta(\infty) \) and \( \tau \) are respectively the asymptotic volume fraction and the compaction characteristic time. This compaction characteristic time is expressed in number of taps. The parameter \( \eta(\infty) \) corresponds to the maximal packing fraction reachable by tapping the powder. As the characteristic tap number \( n_{1/2} \) is obtained directly from the compaction curve and the second one \( \tau \) is the result of the fit by a theoretical law. Therefore, if the theoretical law correctly fits the experimental data, the parameter \( \tau \) does not depend on the number of taps applied on the sample to perform the measurement. One should note that both dynamical parameters are linked by the relation \( n_{1/2} = (e - 1)\tau \).

2.3. Rotating drum measurement

Experimentally, the most practical geometry to study the flow of granular materials is the rotating drum. This flow geometry has been extensively studied [43–45,36], in particular with non cohesive granular materials. In this non-cohesive case, the flow dynamics as a function of the rotating speed \( \Omega \) is well known. At very low angular velocities \( \Omega \), the flow is intermittent. The slope of the pile evolves between the angle of repose \( \alpha_r \) and the maximum angle of stability \( \alpha_m \). At higher velocities, the flow becomes continuous. In this continuous regime, the interface shape of the free surface is essentially flat for low values of the Froude number \( F_r = R \Omega^2/\rho g \), where \( R \) is the drum radius and \( g \) the gravitational acceleration. This flow regime is presented in Fig. 6(a). When the inertial effects become important, a well known S-shape is observed [45]. Finally, when the Froude number \( F_r \) is higher than the unity, a centrifugation is observed. In the case of cohesive grains, the behavior of the granular assembly is more complex. In particular, the continuous regime does not exist. Indeed, the cohesion induces intermittencies in the whole range of rotating speed [46,47]. The flow of a cohesive powder in a rotating drum is presented in Fig. 6(b).

The experimental setup (see Fig. 7) is a horizontal aluminum cylinder of diameter \( D = 84 \) mm and length \( L = 20 \) mm with glass side walls. The drum is half-filled with the powder. The cylinder rotates around its axis at an angular velocity \( \Omega \) producing the flow of the grains. To perform the measurements, the rotating drum is backlighted and recorded with a CCD camera. For each angular velocity, 50 images of the drum separated by 0.5 s are recorded. On the images, the granular material appears in black while the air appears in white. Then, the position of the air/powder interface is determined by an edge detection. The average interface position and the fluctuations around this average position are computed. From the fluctuations of the interface, the standard deviation \( \sigma_{fl} \) is calculated. This parameter is directly related to the cohesion inside the drum. Indeed, in the range of considered rotating speed \( \Omega \), a non-cohesive granular material leads to a continuous flow. From the average interface position, the flowing angle \( \phi_f \) is measured in the center of the flow.

In our study, the rotating drum speed range is selected in order to obtain a flat continuous flow with a non cohesive granular material (see Fig. 6(a)). Therefore, with a non-cohesive granular material, the flow can be fully characterized with the flowing angle \( \phi_f \). The second parameter \( \sigma_{fl} \) enables to quantify the cohesion inside the powder.

The rotating drum allows to measure the flow properties of a powder for different rotating speeds \( \Omega \). Therefore, the flowability is measured as a function of the shearing rates and rheological properties like shear thinning or shear thickening can be evaluated.
2.4. Measurement uncertainties

In order to estimate the uncertainties on the parameters obtained with the three powder characterization methods, the measurements have been repeated five times with a non cohesive and a cohesive granular material. The cohesive material is a silicon carbide abrasive with an average grain size of 13 μm. The non-cohesive material is also a silicon carbide abrasive but with larger grains (250 μm). The detailed analysis of these materials is presented in the next section. The standard deviations have been calculated. Table 2 presents the estimation of the uncertainties for each parameter in percentage of the results.

3. Typical studies

3.1. Influence of the grain size

Silicon carbide abrasives of different grades have been analyzed with the three characterization techniques. The selected grades cover a wide range of grain size \( d \): 250, 170, 100, 80, 50, 30, 23, 13 and 5 μm. Some SEM micrographs of the grains are presented in Fig. 8. The grains are angular, hard and monodisperse. The main characteristic that differentiates these powders is the grain size \( d \). Therefore, this analysis allows to evaluate the influence of the grain size on the physical properties of the assembly.

Fig. 9 shows the evolution of the macroscopic parameters obtained with the GranuHeap measurement. The repose angle \( \alpha_r \) and the deviation \( \sigma_r \) from the ideal heap are presented as a function of the grain size \( d \). Both parameters \( (\alpha_r \text{ and } \sigma_r) \) decrease with the grain size in the range \( d < 50 \mu m \). Afterward, they are constant. The same behavior is observed with the dynamical measures \( (\alpha_f \text{ and } \sigma_f) \) obtained with the rotating drum (see Fig. 11). Since \( \alpha_r \) and \( \sigma_r \) are related to the cohesion inside

![Fig. 8](image1.png)

**Fig. 8.** SEM micrographs of the silicon carbide abrasives. The micrographs are corresponding respectively to the average grain size of (a) 250 μm, (b) 100 μm, (c) 30 μm, and (d) 5 μm.

![Fig. 9](image2.png)

**Fig. 9.** Evolution of the repose angle \( \alpha_r \) and of the deviation \( \sigma_r \) from the ideal heap as a function of the grain size \( d \) for the silicon carbide abrasives. The measurements have been done with the GranuHeap instrument.

![Fig. 10](image3.png)

**Fig. 10.** Evolution of the parameters obtained with the GranuPaQ instrument (the initial \( \eta_0 \) and final \( \eta_f \) packing fractions, the Hausner ratio \( H_r \) and the compaction characteristic time \( n_{1/2} \)) as a function of the grain size \( d \) for the silicon carbide abrasives.
the granular material, we can conclude that the cohesion plays an important role when the grain size becomes lower than 50 μm. Above $d = 50 \mu m$, the cohesion is negligible ($\sigma_r = 0$ and $\sigma_f = 0$). This effect can also be seen on the SEM micrographs (see Fig. 8(d)). Indeed, the smallest grains are forming agglomerates due to the cohesion between the grains.

This behavior is confirmed by the compaction measurements (see Fig. 10). Indeed, the initial and the final packing fractions increase with the grain size in the range $d < 50 \mu m$ till a constant value when $d > 50 \mu m$. For small grains ($d < 50 \mu m$), the cohesive forces between the grains are able to stabilize a very loose packing. When the grain size increases, the ratio between the weight of one grain and the cohesive force acting on this grain increases. Therefore, the cohesive forces become unable to stabilize a loose packing. Moreover, when the initial packing is loose, the tapping mechanism is able to modify the structure of the assembly. Then, for cohesive granular material, the difference between the initial and the final packing fraction is higher. This effect is reflected by the evolution of the Hausner ratio as a function of the grain size $d$. Indeed, $H_r$ decreases as a function of the grain size in the range $d < 50 \mu m$ till a constant value when $d > 50 \mu m$.

The compaction characteristic time $n_{1/2}$ shows a more complex behavior. The decrease of $n_{1/2}$ as a function of $d$ in the cohesive range $d < 50 \mu m$ is expected. The cohesive forces slow down the compaction dynamics. The increase of $n_{1/2}$ as a function of the grain size in the

Fig. 11. Evolution of the flowing angle $\alpha_f$ and of the deviation $\sigma_f$ from the steady flow as a function of the grain size $d$ for the silicon carbide abrasives. The measurements have been made with the GranuDrum instrument.

Fig. 12. SEM micrographs of the flours: (a) Anco fermentante, (b) Anco patisserie, (c) Francine complete, (d) Francine all purpose and (e) Francine fluid.
range $d > 50 \mu m$ is due to the fact that more energy is needed to move a grain when the grains are larger and heavier. Moreover, when the size of the grain increases, the effect of the wall becomes more important. This geometrical effect could also explain the increase of $n_{1/2}$.

All the measurements performed with the silicon carbide abrasives show that the granular materials should be split in two categories. Indeed, when the grain size is lower than 50 $\mu m$, the cohesive forces inside the packing become larger than the other interactions. Of course, the exact value of the grain size corresponding to the transition between a non cohesive to a cohesive granular material will change as a function of the nature of the grains and as a function of the humidity [19–21]. However, this value of 50 $\mu m$ can be used as practical criteria to determine if the cohesion will play a relevant role.

3.2. Influence of the grain size distribution

Except some particular cases like abrasive material, many powders are made of grains from different sizes. Therefore, the grain geometry cannot be characterized by only one number. From the grain size distribution, many parameters can be extracted: the average grain size, the polydispersity, ... In order to study the link between the grain size distribution and the macroscopic properties of powders, five types of flour available in supermarkets have been analyzed. Two flows are coming from the company Anco: “fermentante” and “patisserie”. Three flows are coming from the company Francine: “all purpose”, “complete” and “fluid”. Some SEM micrographs of the grains are presented in Fig. 12. Fig. 13 shows the grain size distributions for all the samples obtained in a dry way with a laser diffraction particle size analyzer (Malvern, Mastersizer Sirocco 2000). All the distributions are bi-modal. A first maximum is observed around 20 $\mu m$ and a second one around 100 $\mu m$. A group of three flows exhibits very similar size distributions (Anco ferm., Anco pat. and Francine all purpose). Two flows are strongly different from this group: Francine fluid and Francine complete. Some descriptors extracted from the grain size distribution are presented in Table 3.

The analysis of the abrasive powder (see the previous section) has shown that the cohesive forces strongly influence the macroscopic properties when the grain size is small. We have shown that for powders, small means a size inferior to around 50 $\mu m$. Therefore, the smallest grains are expected to influence the behavior of a polydisperse powder. In order to measure the influence of the smallest particles, the results of the measurements are considered as a function of the smallest grain size $d(0,1)$. If this hypothesis on the strong influence of the smallest grains is correct, the static and dynamic angle $\alpha_s$ and $\alpha_d$, the static and dynamic cohesive index $\sigma_s$ and $\sigma_d$, the Hausner ratio $H_r$ and the compaction characteristic time $n_{1/2}$ should decrease with $d(0,1)$. Moreover, the initial and the final packing fraction should increase with $d(0,1)$.

Fig. 14 presents the results obtained with the GranuHeap. The decrease of the deviation $\sigma_d$ from the ideal heap shape as a function of $d(0,1)$ shows that the cohesion inside the flour depends on the smallest grain size. This observation is confirmed by the analysis of the flow fluctuations $\sigma_f$ with the GranuDrum method (see Fig. 16). As expected, the static angle $\alpha_s$ and the flowing angle $\alpha_d$ decrease with the smallest grain size $d(0,1)$. One exception is observed with the “Francine fluid” flour. For this flour, the parameters $\sigma_f$ and $\sigma_d$ corresponding to the cohesion are low but the flowing angle $\alpha_d$ is higher than the flowing angle of the other flows. This exception to the general trend shows that only one measurement is not sufficient to characterize the flowability of a powder. The results obtained with the GranuPaq (see Fig. 15) are in agreement with the other observations. Indeed, both compaction ratio $H_r$ and compaction characteristic time $n_{1/2}$ decrease with $d(0,1)$.

Table 3

<table>
<thead>
<tr>
<th>Flour name</th>
<th>$d(0,1)$</th>
<th>$d(0.5)$</th>
<th>$\sigma$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Francine fluid</td>
<td>22.7</td>
<td>136</td>
<td>67.1</td>
</tr>
<tr>
<td>Anco fermentante</td>
<td>17.9</td>
<td>95.5</td>
<td>51.0</td>
</tr>
<tr>
<td>Francine all purpose</td>
<td>16.2</td>
<td>100.6</td>
<td>53.5</td>
</tr>
<tr>
<td>Francine complete</td>
<td>15.6</td>
<td>109.3</td>
<td>105.5</td>
</tr>
</tbody>
</table>

Fig. 13. Grain size distributions obtained with a laser diffraction particle size analyzer (Malvern, Mastersizer 2000) for the samples of flour.

Fig. 14. Evolution of the repose angle $\alpha_s$ and of the deviation $\sigma_d$ from the ideal heap as a function of $d(0,1)$ obtained with the five samples of flour. The measurements have been made with the GranuHeap instrument.

Fig. 15. Evolution of the parameters obtained with the GranuPaq instrument (the initial $\eta_0$ and final $\eta_f$ packing fractions, the Hausner ratio $H_r$ and the compaction characteristic time $n_{1/2}$) as a function of $d(0,1)$ obtained with the five samples of flour.
The heap shape analysis is not shown in this case. Tapped density test, rice grains with different elongations have been made with the GranuDrum instrument. (see Fig. 17). Afterward, the images have been binarized and the black one hundred grains has been scanned with a classical of the SEM micrographs (Fig. 12). Indeed, the small grains are sticking on the big ones and are able to form some cohesive bridges between them. Therefore, the size of the smallest grains is a key parameter for the characterization of a powder.

3.3. Influence of grain shape

For small grains, i.e. for grain diameters inferior to 50 μm, the macroscopic properties of a pile are mainly related to the cohesive forces between the grains. For larger grains, the particle shape becomes an important parameter. In order to show the influence of the particle shape on the macroscopic parameters obtained from the analysis of the flow in the drum and the compaction dynamics during the tapped density test, rice grains with different elongations have been considered. The heap shape analysis is not shown in this case.

To analyze the shape of the rice grains, five samples of approximately one hundred grains has been scanned with a classical office scanner (see Fig. 17). Afterward, the images have been binarized and the black shapes corresponding to the grains have been fitted with ellipses. This simple method allows to obtain the major a and the minor b axes for each grain. The result of this analysis is presented in Table 4. From the “Risotti” to the “Basmati” rice, the elongation a/b evolves between 2.2 and 3.8. The “Long rice No 1” is particular because it contains a lot of broken grains. Therefore, the standard deviation of the long axis a is much larger than the other types of rice.

![Fig. 16.](Image) Evolution of the flowing angle αℓ and of the deviation σℓ from the steady flow as a function of d(0.1) obtained with the five samples of flour. The measurements have been made with the GranuDrum instrument.

![Fig. 17.](Image) Analysis of the rice grain shape. (left) A sample of grains is scanned with a classical office scanner. (middle) The image is binarized to obtain black shapes corresponding to the grains. (left) The black spots are fitted by ellipses to obtain the major a and the minor b axes for each grain.

![Fig. 18.](Image) Evolution of the parameters obtained with the GranuPaQ instrument (the initial η0 and final η∞ packing fractions, the Hausner ratio Hr and the compaction characteristic time t(1,2)) as a function of the grain elongation a/b for five samples of rice.

Table 4

<table>
<thead>
<tr>
<th>Rice</th>
<th>a [mm]</th>
<th>b [mm]</th>
<th>a/b</th>
</tr>
</thead>
<tbody>
<tr>
<td>Risotti</td>
<td>6.1±1.1</td>
<td>2.8±0.6</td>
<td>2.2</td>
</tr>
<tr>
<td>Long rice No 1</td>
<td>6.0±1.5</td>
<td>2.2±0.3</td>
<td>2.7</td>
</tr>
<tr>
<td>Bosto Wok</td>
<td>6.7±1.0</td>
<td>2.4±0.3</td>
<td>2.8</td>
</tr>
<tr>
<td>Thai</td>
<td>6.7±1.4</td>
<td>2.2±0.3</td>
<td>3.0</td>
</tr>
<tr>
<td>Basmati</td>
<td>7.3±1.1</td>
<td>1.9±0.1</td>
<td>3.8</td>
</tr>
</tbody>
</table>

Fig. 18 shows that the elongation a/b influences the packing fraction and the compaction dynamics. The decrease of the packing fraction as a function of the elongation of the grains is a well know effect [17]. Indeed, when the grains are longer, some large voids are present in the pile. Moreover, when the elongation a/b increases, the mobility of the grains inside the packing decreases due to steric frustrations [48]. This effect explains the increase of the compaction characteristic time with the elongation (see Fig. 18). The point that does not follow the general trends corresponds to the “Long rice No 1” which has a wider grain length distribution due to the presence of broken grains.

As shown by Fig. 19, the flowing angle αℓ and the fluctuations of the flowing interface σℓ increase with the grain elongation a/b. In this case, the fluctuations of the flow αℓ is related the elongation of the grains, not to cohesive forces. Indeed, grain elongations are known to induce intermittencies in flows [49]. The flowing angle αℓ is found to increase with the drum rotating speed Ω. This is a classical behavior with non cohesive granular materials. This tendency has not been observed with the abratives and with the flours probably due to an effect of rheofluidization.

4. Conclusion

Three well known powder characterization tests have been optimized in the light of recent fundamental research on granular materials. In order to illustrate how these measurements could be used in practical cases, analyses are performed with three types of granular materials: silicon carbide abrasives, flours and rice. The macroscopic analysis of the mono-disperse abrasives of different grades has shown that the cohesion between the grains becomes important when the grain diameter is inferior to 50 μm. Moreover, the cohesion modifies the static, the quasi-static and the dynamic properties of the pile. Indeed, the cohesion
strongly decreases the packing fraction of the pile and increases the Hauser ratio $H_r$ and the compaction characteristic time $t_{1/2}$. The angle of repose $\alpha_1$ and the flowing angles $\alpha_f$ are also increased by the cohesion. In addition, the cohesion induces some fluctuations of the flow and some irregularities at the surface of a pile. Concerning the polydisperse powders, the analysis of different flows has shown that the smallest grains influence the macroscopic properties of the powder. For larger grains $d > 50 \mu$m, the particle shape becomes an important parameter. In particular, the elongation of the grain influences the macroscopic properties of the assembly.

Acknowledgments

GL thanks F.R.S.-FNRS, FB thanks the Walloon Region (First SO no 071/6576) and KT thanks the Walloon Region (BIOFINA Convention no 6045) for the financial support. This work has also been supported by INANOMAT project (JAP P6/17) of the Belgian Science Policy. Thanks to Catu (microscopy center of the University of Liège) for the SEM micrographs.

References

[1] Number of records obtained in the database Web of Science of Thomson Reuters. The search has been made with the keywords “Granular material” OR “Granular materials” in the Topic.