



# Ambivalent role of fine particles on the stability of a humid granular pile in a rotating drum

Xixi Huang, Sandrine Bec, Jean Colombani

## ► To cite this version:

Xixi Huang, Sandrine Bec, Jean Colombani. Ambivalent role of fine particles on the stability of a humid granular pile in a rotating drum. Powder Technology, 2015, 279, pp.254-261. 10.1016/j.powtec.2015.04.007 . hal-02308423

**HAL Id: hal-02308423**

**<https://hal.science/hal-02308423>**

Submitted on 17 May 2022

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

# Ambivalent role of fine particles on the stability of a humid granular pile in a rotating drum

Xixi Huang<sup>a,b</sup>, Sandrine Bec<sup>b</sup>, Jean Colombani<sup>a,\*</sup>

<sup>a</sup>*Institut Lumière Matière; Université de Lyon; Université Claude Bernard Lyon 1; CNRS UMR 5306; Domaine scientifique de la Doua, F-69622 Villeurbanne, France*

<sup>b</sup>*Laboratoire de Tribologie et Dynamique des Systèmes; Université de Lyon; École Centrale de Lyon; CNRS UMR 5513; 36, av. Guy de Collongue, F-69134 Écully, France*

---

## Abstract

We have studied the influence of fine particles on the stability of a granular medium in a rotating drum. The stability diagram of this system was established as a function of the drum rotation speed, fine particle content and relative humidity. Four regimes were observed. At low fine content, an avalanching regime is encountered at low rotation rate, and a continuous flow regime at high rotation rate. At high fine content, a stick-slip regime at the drum wall is seen at low rotation rate and a continuous sliding regime at high rotation rate. The influence of fines is ambivalent. At low fine content, they fluidize the pile and decrease its stability, by granular lubrication. At high fine content, they solidify the pile and increase its stability, by jamming the grain assembly. The enhancement of humidity increases the stability for high fine content, but has no effect for low fine content.

*Keywords:* stability, rotating drum, dust, humidity, lubrication, jamming

---

## 1. Introduction

Granular matter is one of the most complicated materials, as it can present either gaseous, liquid and solid behaviors. Its mechanical properties have been studied by various methods : inclined plane, tilted box, rotating drum, etc. Among these configurations, the rotating drum is commonly used in laboratory experiments and also in the industry for processing granular matter [1]. Indeed, in the simple configuration of a rotating cylinder, several processes can be performed : mixing, drying, segregation or chemical reactions.

---

\*. jean.colombani@univ-lyon1.fr, +33 472448570

The stability of a dry or humid granular pile in a rotating drum has been extensively studied, revealing the richness of the granular motion inside the cylinder. By varying the rotation velocity, different regimes of the bed behavior are observed : slipping, slumping, rolling, cascading, cataracting and centrifuging. Among them, slumping and rolling are usually used in industry for material mixing [2]. At low rotation velocity, the granular pile is lifted as a solid body pursuing the rotating drum, the surface angle  $\theta$  of the pile increases until a maximum angle of stability, the "upper repose angle", named  $\theta_m$ , is reached, then the granular pile destabilizes at its surface and particles on top start to roll downhill as an avalanche. When the slope of the pile reaches a minimum angle of stability, the "lower repose angle", called  $\theta_r$ , the avalanche stops, and so on until another avalanche occurs. This regime is defined as the slumping or discrete avalanche regime. The subsequent rolling regime occurs when the rotation velocity is increased. In this case, the cycle duration is shorter than the avalanche duration. Thereby the grains at the surface continuously roll, keeping the surface angle constant.

A lot of researches have studied the granular bed behavior in the slumping and rolling regimes. However most of these studies are performed with a monodisperse granular matter, while in industrial processes the granular material is usually bidisperse or even polydisperse. Moreover even a so-called monodisperse granular medium can contain a certain quantity of small particles due to the wear of the grains, especially in the grinding process [3]. The presence of such "dust" particles of very small size is one of the origins of the phenomenon of re-agglomeration and caking of particles [4, 5]. Up to now, it has hardly been studied in experimental researches.

By adding a small quantity of fine particles to a monodisperse granular pile and measuring the maximum stability angle of this pile in a rotating drum with a controlled relative humidity, we have shown recently that the influence of the fines on the pile stability is ambivalent [6]. We detail here the experiments leading to this result and show how, at low fine content, the small particles play the role of granular lubricant and make the heap more unstable, and at higher content, they block the inner motions inside the heap and solidify it. These results enable to make a link between independent and inconsistent observations, that had not been compared until now, showing that

fine particles in one well-defined system may either play a fluidizing or a jamming role, depending on their percentage.

## 2. Experiments

### 2.1. Experimental setup

The experimental setup is illustrated in figure 1, based on a device used in previous studies [4]. The granular material is introduced in a stainless steel drum of 10 cm radius and width, rotating around its horizontal axis at a fixed speed controlled by a synchronous electric motor (AKM23D AN CNAA, Kollmorgen). The rotation speed of the cylinder can be varied from 0.0001 to 500 rpm. In this study only slow rotation speeds were used. The cylinder is enclosed by two glass windows with a hole at their center for air exchange with the outside atmosphere. The drum is partially filled with a fixed quantity of 200 g of glass beads, **with a filling ratio of 30%**. The glass beads used in this study are soda-lime glass beads (BV250) of diameter  $(250 \pm 50) \mu\text{m}$  (Sigmund Lindner). The surface of the glass beads has been observed by atomic force microscopy (AFM) and scanning electron microscopy (SEM), showing a small roughness of the surface, typically 500 nm (figure 2). Fine glass beads (BF10) of diameter  $(5 \pm 4) \mu\text{m}$  of the same material are added in the granular pile as fine particles. **The density of the soda-lime glass is 2.50 kg/L and the bulk density of the bed of large particles is 1.46 kg/L (manufacturer data).**

**Usual bidisperse granular mixtures are known to segregate. Therefore we have sampled the mixture at the top and bottom of the heap at the end of some experiments and we have observed by optical microscopy that the fine particles content was uniform. This absence of segregation finds an explanation in the characteristic distribution of the fine particles among the large ones (see section 4.1 and figure 13).**

The experimental setup is placed in a sealed poly(methyl methacrylate) box. Both relative humidity and temperature are controlled in the experiments. The temperature in the box is controlled with a heating resistor regulated by a temperature controller, and is fixed at  $29.0^\circ\text{C}$ . A large flat beaker of saturated aqueous inorganic salt solution is placed in the box to maintain a constant

value of relative humidity during the experiments. Four different salts are used in our experiments. Table 1 shows the theoretical value of the relative humidity in the atmosphere in the presence of the saline solutions at 25°C. The relative humidity  $H$  is measured by a hygrometer with an accuracy of 1.5%. As the experiments are carried out at 29°C, and a slight discrepancy may exist between the expected and real values of the relative humidity, only measured values are used thereafter. A fan is placed inside the box to ensure homogeneous temperature and humidity.

The granular pile is illuminated from the front through the glass windows to improve the surface detection (figure 3). A digital video camera placed in front of the drum is used to continuously record the behavior of the granular medium. The angle of the surface of the pile with the horizontal is automatically calculated from the captured images. The uncertainty in the angle measurements is typically  $\pm 0.5^\circ$ .

## 2.2. Procedure

1. A 200 g mixture of coarse and small beads is mixed in a vessel by hand.
2. The mixture is introduced in the drum in rotation at relatively low speed (1 rpm) for 24 h in the desired environment to homogenize the atmosphere throughout the granular pile.
3. Rotation at 5 rpm during 30 s provides an initial reference state of the pile.
4. Rotation at the desired speed  $n$  during a few cycles is used as "pre-measurement". It permits to determine the flowing regime of the granular medium. If the regime is avalanching or stick-slip (see below), it gives an indication of the maximum and minimum angles of stability  $\theta_m$  and  $\theta_r$  of the granular pile. Figure 4 shows an example of pre-measurement in the avalanche regime. If the regime is continuous flowing or sliding (see below), it gives an indication of the dynamic repose angle. The reference angle  $\theta_{ref}$  is determined as the angle  $0.5^\circ$  smaller than the maximum angle or dynamic repose angle.
5. The drum rotates at the same rotation speed  $n$ , and the evolution of the surface angle of the granular pile  $\theta$  is recorded versus time. For each turn, a 3 s stop, which corresponds to the waiting time  $t_w$ , is effected at  $\theta = \theta_{ref}$ . Then the rotation continues in the same direction at

the same speed until an avalanche occurs. The avalanche angle  $\theta_m$  and repose angle  $\theta_r$  are recorded (figure 4).

6. The last step is repeated 100 times and the values of the avalanche angle  $\theta_m$  and repose angle  $\theta_r$  are determined from the average value of the 100 measurements **performed by automatic image analysis**.

The most important experimental quantities in this procedure are the waiting angle  $\theta_{ref}$  and waiting time  $t_w$ . Indeed previous studies have shown that the stability of the heap firmly depends on these two values. This influence is a consequence of the fact that the cohesion of the heap partly stems from the slow condensation of capillary bridges between the beads [4]. So the maximum stability angle is observed to enhance when the waiting time or waiting angle is increased. For this reason, all experiments were carried out with the same  $\theta_{ref}$  and  $t_w$  values.

Based on this procedure, three major parameters affecting the stability of the strongly bidisperse granular pile in the rotating drum were varied : (a) the volume fraction  $\phi$  of fine particles ; (b) the rotation speed  $n$  ; (c) the relative humidity  $H$ .

### 3. Results

#### 3.1. Stability diagram

Figure 5 shows the diagram of stability of the granular pile obtained by varying the fraction of fines  $\phi$ , the rotation speed  $n$  and the relative humidity  $H$ . **In this diagram, each of the 243 experiments performed for each humidity is represented by a dot. By varying  $n$  and  $\phi$ , four different flowing regimes are observed : avalanching, continuous flowing, stick-slip and continuous sliding. Line segments separate the dots pertaining to each regime, sharing the diagram in four rectangles. When these separation lines depend on humidity, their position for each humidity is drawn.**

For each condition of relative humidity, at small concentration of fine particles **(lower part of the diagram)**, the avalanche regime is observed at low rotation speed  $n$ . When the drum is rotating faster than  $\sim 1$  rpm, the regime of continuous flow occurs. When  $\phi$  increases and reaches a critical

value  $\phi_c$ , the behavior of the granular medium changes : a phenomenon of stick-slip at the inner wall of the drum is observed and alternates with the avalanche regime. The two regimes coexist when  $\phi$  is between 0.15 and 0.24%. For a larger fine fraction (upper part of the diagram), the surface of the pile stabilizes, no more avalanche is observed and only the stick-slip behavior at the drum wall is present. In this configuration, if  $n$  is increased, the stick-slip regime transforms in a continuous sliding behavior.

### 3.2. Low volume fraction of fines

When  $\phi < \phi_c$ , the destabilization of the particles occurs at the surface of the granular heap. The transverse flow behavior of granular matter has been studied widely in the literature [7, 8], depending on several parameters, principally the rotation speed and the filling level. Here, as we use a rotation speed below 10 rpm and a fixed filling level, only the longitudinal avalanche and continuous flow regimes will be discussed.

#### 3.2.1. Rotation speed influence

In the avalanche regime, during the rotation of the drum, avalanches of several layers of particles detach from the bed and slumps toward the lower extremity of the bed surface. The slump occurs periodically during the rotation, each time the surface reaches the maximum angle of stability  $\theta_m$  (the upper angle of repose). Following a slump, the bed inclination reaches a steady position at a minimum angle of stability  $\theta_r$  (the lower angle of repose) (figure 6). When the rotation speed increases ( $> 1$  rpm), the intermittent avalanches turn to a continuous flow, which is characterized by a continuous rolling of a few layers of particles at the bed surface. In the rolling regime, the surface of the granular pile appears flat with a constant angle of inclination, defined as the dynamic repose angle  $\theta_{dyn}$  (figure 6). The measurements of  $\theta_m$ ,  $\theta_r$  and  $\theta_{dyn}$  are carried out for  $n$  ranging from 0.05 to 10 rpm.

An example of the evolution of the upper  $\theta_m$ , lower  $\theta_r$ , and dynamic  $\theta_{dyn}$  repose angles versus rotation speed is shown in figure 7. In the avalanche regime,  $\theta_m$  and  $\theta_r$  exhibit an approximately constant value when  $n$  is increased. The only influence of the rotation speed here is the duration

between two consecutive avalanches which shortens when the rotation speed increases [9]. As we maintain the heap immobile at an angle slightly below  $\theta_m$  during a constant waiting time before each avalanche (figure 4), the state of cohesion is always identical [10], so the maximum stability angle does not vary. In the continuous flow regime, the dynamic repose angle rises linearly with  $n$  (figure 7), as expected numerically [1]. The transition between both regimes occurs when the rotation becomes too rapid for the surface to have time to relax from  $\theta_m$  to  $\theta_r$ . For instance in figure 7, the transition occurs at  $n = 0.7 \text{ rpm} = 4.2^\circ/\text{s}$ . We see that at that point,  $(\theta_m - \theta_r) = 2.7^\circ$  and the avalanche duration has been measured as  $\tau = 593 \text{ ms}$ , corresponding to  $n_c = \frac{\theta_m - \theta_r}{\tau} = 4.55^\circ/\text{s}$ , in accordance with the observed value.

The transitions between regimes in granular media in a rotating drum have been studied since the 1980s with different geometries, i.e., rotating drum and rectangular box [8, 11, 12]. Rajchenbach observed that this transition is dependent upon the direction of variation of the rotation speed, the transition showing an hysteresis [8]. Fischer et al. showed later that it was rather a system with intermittent erratic transitions between the two modes [13]. In our work, all experiments were performed with increasing rotation speed, gradually increasing from 0.05 rpm to 10 rpm. As Fischer et al., we obtain that during the transition of regimes, both avalanches and continuous flow exist intermittently during the rotation of the drum (figure 6).

### 3.2.2. Fine particles influence

In the diagram of stability, it can be noticed that the influence of fine particles on the flow regime is surprisingly significant, even though the volume fraction  $\phi$  is relatively low. Concerning the avalanche regime (when  $\phi < 0.15\%$ ), one can see in figure 5 that when the fine concentration  $\phi$  increases, the transition between avalanche and continuous flow is shifted toward lower rotation speeds. Besides, the measured repose angles  $\theta_m$  and  $\theta_r$  decrease with the increase of fine volume fraction  $\phi$  (figure 8). Similar results have been observed in the rolling regime, illustrated in figure 9, where the dynamic repose angle decreases when the fine content increases. These three results all show a fluidization of the surface by the dust, the heap being less and less stable when  $\phi$  enhances. This feature is emphasized in the  $\phi$ - $\theta_m$  plot of figure 10.



### 3.2.3. Relative humidity influence

As shown in figure 10, in the avalanche regime, there is no significant influence of the relative humidity on the maximum angle of stability since the four series of data are superimposed.

## 3.3. High volume fraction of fines

### 3.3.1. Rotation speed influence

Once the fine particles concentration is higher than 0.24%, the granular pile exhibits a total solidification. It slips at its bottom against the drum wall as a quasi-solid body. This behavior is called the stick-slip regime. This regime had never been observed before in granular media in a rotating drum.

In the stick-slip regime, the maximum and minimum angles are measured in a similar way as in the avalanche regime. When the drum rotates, the pile remains stuck to the wall until a maximum stability angle is reached ( $\theta_{\text{mg}}$ ), then it detaches and slips downward to the minimum angle ( $\theta_{\text{rg}}$ ) without any granular motion at the surface. The only observation of granular motion is seen in a layer of grains at the contact between the pile and the drum wall. When the rotation rate increases, the intermittent stick-slip transforms into a continuous sliding of a thin layer of grains at the pile/drum wall contact. As in the avalanche regime, this arises from the fact that the cycle duration becomes shorter than the sliding duration. The surface angle of the granular pile ( $\theta_{\text{ro}}$ ) remains now stable during the rotation. Figure 11 shows that varying the rotation speed has **only a slight impact** on the three angle values.

### 3.3.2. Fine particles influence

The influence of the fine particles on the maximum angle of stability  $\theta_{\text{mg}}$  is now studied in the stick-slip regime. Figure 12 illustrates the evolution of  $\theta_{\text{mg}}$  when  $\phi$  increases from 0.2% to 1%. Unlike the avalanche regime, the maximum angle of stability is enhanced by the presence of fine particles. So whereas in the avalanche regime the pile was fluidized by the fines, it is stabilized in the stick-slip regime.

### 3.3.3. Relative humidity influence

As shown in figure 12, the relative humidity has an influence on the maximum angle of stability  $\theta_{\text{mg}}$ . So the influence of fines in this regime is completely different from the case of avalanche regime, where humidity has no observable influence on the pile stability.

These experimental results show a rich set of, sometimes opposing, behaviors. In the next section we will try to propose a microscopic interpretation and a quantitative modeling of the most interesting effects : the fine content and humidity influences on the pile stability.

## 4. Discussion

### 4.1. Avalanche regime

When a small quantity of fine particles is added to the granular pile, a fluidizing effect on the surface of the pile is observed (figure 10).

In the analysis of Mohr-Coulomb, a granular pile loses its stability in the plane situated at depth  $h$  where the ratio of the shear stress  $T$  (due to the weight  $W$  of the above grains) and the normal stress  $N$  (due to the weight  $W$  of the above grain and to the cohesion strength  $c$ ) is greater than a friction coefficient  $\mu$ .

Between two beads of a pile placed in an unsaturated humid environment, the water wettability on glass lowers the chemical potential of liquid water compared to the chemical potential of water vapor. Therefore below a critical distance, of the order of a few nanometers, the steam becomes metastable and capillary bridges may nucleate. As the grain surface is rough at the nanometer scale (figure 2), the actual contact is reduced to points at the end of surface asperities [14]. Except for almost saturated humid atmospheres, nucleation of liquid bridges is limited to these asperities and voids exist within the contact [15]. These bridges induce an adhesion strength  $F_{\text{adh}}$  between the grains because of the Laplace pressure.

As evidenced by experiments of surface force apparatus, this adhesion force  $F_{\text{adh}}$  may depend on the normal load applied to the grains [16]. If it is likely not to be the case for flexible materials [17], it should be true for most of hard materials, where the stress cannot be accommodated by

**elastic strain.** Indeed, the burden of the above grains induces plastic deformation of the asperities in contact, leading to a decrease in the gap between the grains. This induces an increase in the number of contact points, so in the number of liquid bridges and in the adhesive force  $F_{\text{adh}}$ . Therefore this adhesion force increases with  $W$ , so with the depth  $h$  of the considered plane. For a large amount of liquid, when all the interstitial space is invaded by the liquid bridges, the effect of reducing the gap between the particles is negligible [18, 19, 20]. But for a small amount of liquid or when the liquid originates from a condensable vapor, it is dominant and it has been shown that in an assembly of mineral spheres, it generally obeys  $F_{\text{adh}} \sim h$  [21]. As  $W = \rho gh$  ( $\rho$  density of the pile and  $g$  acceleration of gravity), we then have both  $W \sim h$  and  $F_{\text{adh}} \sim h$ . In this situation, called the granular regime, the most unstable plane remains the surface [9, 21]. We attribute the experimental observation that our heap always breaks at the surface to this depth-dependent cohesion inside the pile.

When the fine particles are introduced into the granular pile, due to the large diameter ratio ( $d_l/d_s \approx 50$ ), we can assume that the fines feed easily the contacts between the coarse grains and separate them. To check this hypothesis of particle distribution, we have performed SEM observations of the heap just after a rotating drum experiment. We have observed that, beside some fines stuck at the large beads surface, most of them populate the inter-particle contact. An example is shown in figure 13.

This spacing by the fines should induce a decrease of the capillary bonding between large particles. We attribute the fluidization of the surface seen in figure 10 to this granular lubrication of the large grains assembly by the fines<sup>1</sup>. In the granular regime, the maximum stability angle writes :  $\tan \theta_m = \tan \theta_0(1 + C)$  [21]. In this expression,  $\theta_0 = \arctan \mu$  is the maximum stability angle in absence of cohesion and  $C$  a term depending on the adhesion stress inside the pile. The value of  $\theta_0$  has been determined both theoretically and experimentally as being  $23.4^\circ$  in a monodisperse assembly [22]. We see in figure 10 that the minimal value of  $\theta_m$  in this curve, at

---

1. We would like to emphasize that the fluidization here is a consequence of granular lubrication, not of gas injection, as usually in powder handling.

$\phi_c \simeq 0.18\%$ , corresponds to the value of  $\theta_0$ . From this, we deduce that the lubrication of the heap due to the dust has led to the complete destruction of the adhesion inside the heap at  $\phi_c$ . At this point, it behaves as a cohesion-free grain set. We have fitted all the experimental dots in the avalanche regime by the above-mentioned law (figure 10). The agreement is very good for an evolution of the cohesion stress with the fine content of the form :  $C = A(\phi - \phi_c)^n$ , with  $A = 13.5$  and  $n = 2.3$ . This law corresponds in other words to the evolution of the lubrication efficiency with the fine percentage. The satisfactory agreement between the experiments and theory validates our assumption of the lubricating influence of the fines on the pile in the avalanche regime.

The independence of  $\theta_m$  with humidity in this regime can be explained by the fact that in a relatively dry atmosphere ( $H < 60\%$ ), complete capillary bridges between grains do not form. As explained above, only nano-scale capillary condensation occurs at the tip of asperities in the contact zone between two grains [14]. In this situation, the condensates **are responsible for an adhesion force between the beads, but as they remain** always smaller than the asperities, the adhesion force does not vary and the stability does not change with humidity, as already observed in monodisperse media [23].

#### *4.2. Transition between the two regimes*

It has been observed that, at low fine content, the fines play the role of bearing balls between the large grains. In contrary to this lubrication effect, the fine particles show an ability of "jamming" the granular pile in the regime of stick-slip, increasing the cohesion of the pile of coarse grains. This phenomenon of solidification should result from the increase of cohesion between grains inside the granular pile due to the creation of new capillary bridges between fine and coarse particles. As shown in figure 13, fines seem to reform progressively a meniscus bridging their neighbouring particles. This leads, from  $\phi_c$  on, to a complete setting of the assembly.

**We would like to emphasize that this brutal change occurs at a very small, unexpected, value. A change of behavior was expected when the fine particles populate the whole interstitial spaces between the large ones —roughly for  $\phi \sim 40\%$ — thereby beginning to dominate the mixture [24]. But for a value as low as  $\phi_c = 0.18\%$ , the packing factor remains almost unchanged compared to**

the fines-free heap. So the change of stability cannot be linked to the change of filling and must be searched in the preferential distribution of fine beads at the contact between large ones.

#### 4.3. Stick-slip regime

The motion of the granular pile in the stick-slip regime is governed by the friction between the granular assembly and the inner wall of the drum. The maximum stability angle can thus be deduced from a standard **Coulomb** analysis. We propose here such a model to describe the effect of fines.

With the notation introduced in the preceding section, the maximum angle of stability of the pile in the stick-slip regime can be written in a **Coulomb** approach [10, 25] :

$$\tan \theta_{\text{mg}} = \tan \theta_0 \left( 1 + \frac{c}{\rho g h \cos \theta_{\text{mg}}} \right). \quad (1)$$

Here it should be emphasized that  $h$  is the depth of the pile-drum contact plane, so it is the total height of the pile, and that  $c$  is the cohesion stress between the heap and the drum wall.

The more fines are introduced in the granular pile, the more liquid bridges are created between the heap and the drum wall. Thereby an increase of the total adhesion  $c$  between the pile and the drum wall occurs.

The adhesion stress  $c$  is linked to the quantity of fines per unit surface at the drum-pile interface, so to  $\phi^{2/3}$ ,  $\phi$  being the volume fraction of fines. Using the approximation  $\cos \theta_{\text{mg}} \simeq \cos \theta_0$  [25], the maximum stability angle writes :

$$\tan \theta_{\text{mg}} = \tan \theta_0 \left( 1 + \frac{B(\phi - \phi_c)^{2/3}}{\rho g h \cos \theta_0} \right) \quad (2)$$

In this expression, the  $B$  factor, characterizing the cohesion increase due to the fines, is linked to the capillary bridge formation mechanism. Therefore it depends on humidity and on the beads and drum surface roughness. It has been shown that the dependence on relative humidity has the form  $B = D/\ln(1/H)$  [10],  $D$  being a purely geometric parameter. Therefore the maximum stability angle should obey :

$$\tan \theta_{\text{mg}} = \tan \theta_0 \left( 1 + \frac{D(\phi - \phi_c)^{2/3}}{\ln(1/H)\rho gh \cos \theta_0} \right) \quad (3)$$

We have fitted all the experimental results in the stick-slip regime with equation 3. As shown in figure 12, the agreement between the experimental and theoretical curves is very good, thus validating our assumption of the influence of fines on the drum-wall adhesion.

## 5. Conclusion

We have presented here the effect of very fine particles on the stability of a humid granular pile in a rotating drum. Various amounts of fine glass beads of mean size  $5 \mu\text{m}$  (0 to 1% volume fraction) have been added to a granular pile of glass beads of  $250 \mu\text{m}$  mean size rotated in a drum. We have established the stability diagram of this granular medium as a function of fine concentration, relative humidity and rotation velocity. The presence of fine particles has shown an ambivalent effect on the stability of the granular heap. At low fine concentration ( $< 0.18\%$ ), the heap destabilizes through avalanches at the pile surface when the drum rotates, and increasing the fine quantity tends to decrease the upper repose angle  $\theta_m$ . In contrast, once the concentration is more than  $0.24\%$ , the destabilization of the heap proceeds through a stick-slip phenomenon at the drum wall, and the increase of the fraction of fines tends to increase the  $\theta_m$  value.

For large rotation velocities, the avalanche regime evolves into a continuous flow regime, and the stick-slip regime transforms into a continuous sliding regime.

This apparently contradictory behavior is linked to the dual influence of fines on the pile cohesion. In the small fine concentration regime, the fines act as ball bearings and their granular lubrication effect destabilizes the heap. For a higher fine content, the fines induce a jamming of the granular medium, leading finally to an increase of the bulk cohesion of the heap. So in the large fine concentration regime, the heap behaves as a solid body and the destabilization occurs at its bottom. In this case, the fine particles contribute to increase the capillary bonds between the pile and the drum and increases its stability.

We have also studied the influence of relative humidity on the granular stability in our experiments. Again a contradictory behavior is observed. We have found out in the low fine concentration regime that the maximum stability is independent of humidity. On the other hand, in the regime of high content of fines, the maximum stability angle is highly dependent on relative humidity, fines inducing additional capillary condensation between the pile and the drum wall.

Now, these flow regimes in strongly bidisperse granular mixtures have to be investigated with particles made in other materials, to test their universality. As the agglomeration of the grain assembly at high fines content may be detrimental for some applications, like mineral grinding, the search of chemical additives limiting this solidification is another direction of investigation.

### **Acknowledgements**

We thank Elisabeth Charlaix and Lydéric Bocquet for fruitful discussion and the Carnot Institute Ingénierie@Lyon for funding.

## Bibliography

- [1] G. Seiden, P. Thomas, Complexity, segregation, and pattern formation in rotating-drum flows, *Reviews of Modern Physics* 83 (2011) 1323–1365.
- [2] J. M. Ottino, R. M. Lueptow, On mixing and demixing, *Science* 319 (5865) (2008) 912–913.
- [3] L. Opoczky, Fine Grinding and Agglomeration of Silicates, *Powder Technology* 17 (1977) 1 – 7.
- [4] F. Restagno, C. Ursini, H. Gayvallet, E. Charlaix, Aging in humid granular media, *Physical Review E* 66 (2002) 1–7.
- [5] M. Mathlouthi, B. Rogé, Water vapour sorption isotherms and the caking of food powders, *Food Chem.* 82 (2003) 61–71.
- [6] X. Huang, S. Bec, J. Colombani, Influence of fine particles on the stability of a humid granular pile, *Physical Review E* 90 (2014) 052201.
- [7] H. Henein, J. K. Brimacombe, A. P. Watkinson, Experimental Study of Transverse Bed Motion in Rotary Kilns, *Metallurgical Transactions* 14 (1983) 191–205.
- [8] J. Rajchenbach, From Discrete Avalanches to Continuous Regime, *Physical Review Letters* 65 (1990) 2221–2225.
- [9] P. Tegzes, T. Vicsek, P. Schiffer, Avalanche Dynamics in Wet Granular Materials, *Physical Review Letters* 89 (2002) 094301.
- [10] L. Bocquet, E. Charlaix, S. Ciliberto, J. Crassous, Moisture-induced ageing in granular media and the kinetics of capillary condensation, *Nature* 396 (1998) 735–737.
- [11] P. Lemieux, D. J. Durian, From Avalanches to Fluid Flow : A Continuous Picture of Grain Dynamics Down a Heap, *Physical Review Letters* 85 (2000) 4273–4276.
- [12] X. Liu, E. Specht, J. Mellmann, Slumping-rolling transition of granular solids in rotary kilns, *Chemical Engineering Science* 60 (2005) 3629 – 3636.



- [13] R. Fischer, P. Gondret, M. Rabaud, Transition by Intermittency in Granular Matter : From Discontinuous Avalanches to Continuous Flow, *Physical Review Letters* 103 (2009) 2–5.
- [14] D. Tabor, F. P. Bowden, *The Friction and Lubrication of Solids*, Oxford University Press, 1950.
- [15] L. Bocquet, E. Charlaix, F. Restagno, Physics of humid granular media, *C.R. Physique* 3 (2002) 207.
- [16] F. Restagno, J. Crassous, C. Cottin-Bizonne, E. Charlaix, Adhesion between weakly rough beads, *Phys. Rev. E* 65 (2002) 042301.
- [17] J. Lanier, M. Jean, Experiments and numerical simulations with 2d disks assembly, *Powder Technology* 109 (2000) 206–221.
- [18] S. Nowak, A. Samadani, A. Kudrolli, Maximum angle of stability of a wet granular pile, *Nature Physics* 1 (2005) 50–52.
- [19] S. H. Chou, S.S. Hsiau, Experimental analysis of the dynamic properties of wet granular matter in a rotating drum, *Powder Technol.* 214 (2011) 491–499.
- [20] S. S. Hsiau, C. C. Liao, C. H. Tai, C. Y. Wang, The dynamics of wet granular matter under a vertical vibration bed, *Granular Matter* 15 (2013) 437–446.
- [21] F. Restagno, L. Bocquet, E. Charlaix, Where does a cohesive granular heap break?, *The European physical journal. E, Soft matter* 14 (2004) 177–83.
- [22] R. Albert, I. Albert, D. Hornbaker, P. Schiffer, A. Barabasi, Maximum angle of stability in wet and dry spherical granular media, *Phys. Rev. E* 56 (1997) R6271.
- [23] N. Fraysse, H. Thomé, L. Petit, Humidity effects on the stability of a sandpile, *The European Physical Journal B* 619 (1999) 615–619.
- [24] S. Naeini, M. Baziar, Effect of fines content on steady-state strength of mixed and layered samples of a sand, *Soil Dynam. Earthquake Eng.* 24 (2004) 181.

[25] T. Halsey, A. Levine, How Sandcastles Fall, Physical Review Letters 80 (1998) 3141–3144.

TABLE 1: Relative humidity provided by saturated salt solutions at fixed temperature.

Salt	Theoretical relative humidity at 25°C
Lithium bromide (LiBr)	6%
Potassium acetate ( $\text{KCH}_3\text{CO}_2$ )	23%
Potassium carbonate ( $\text{K}_2\text{CO}_3$ )	43%
Sodium bromide (NaBr)	58%

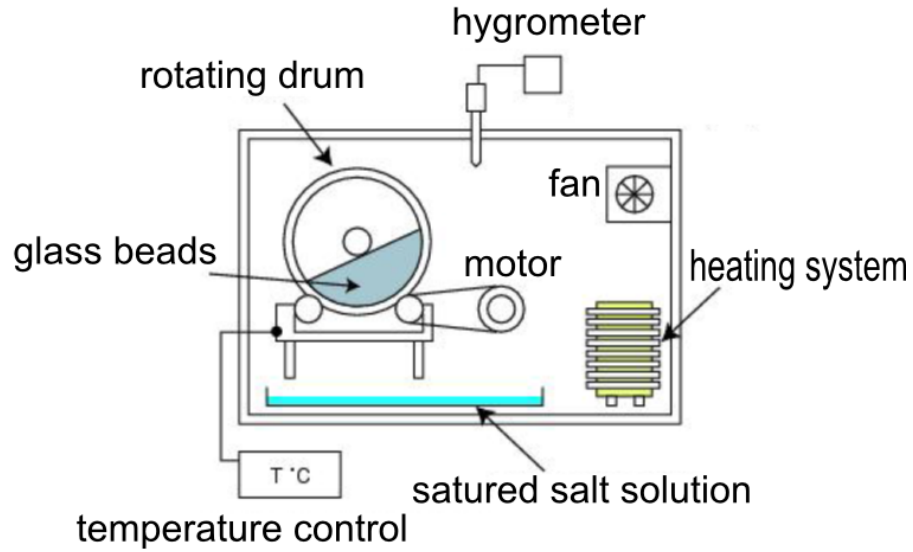


FIGURE 1: Experimental setup.

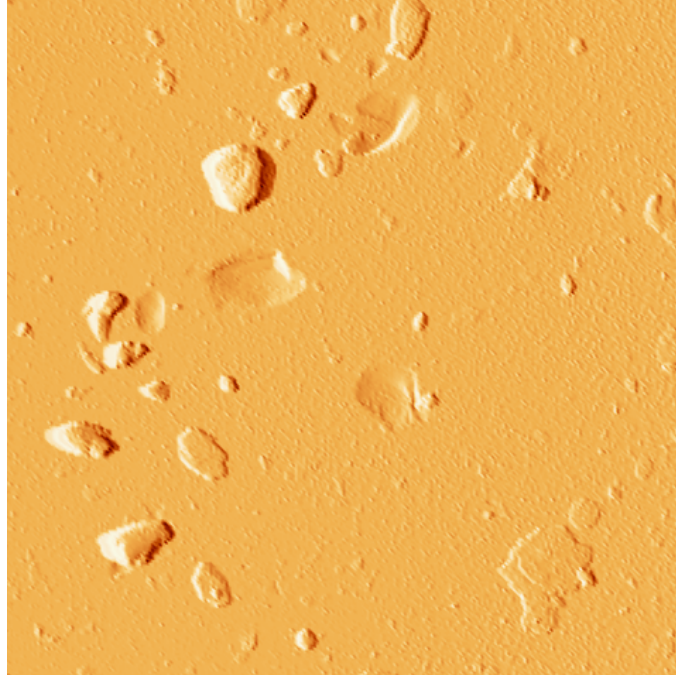


FIGURE 2: AFM observation of the large glass bead surface (error signal). The picture size is  $15 \times 15 \mu\text{m}^2$ .

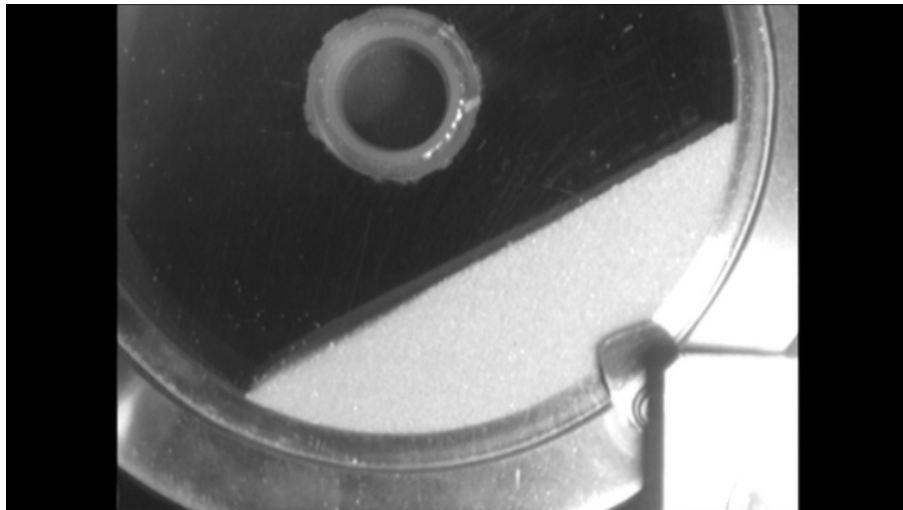


FIGURE 3: Front view of the granular heap inside the 10 cm diameter rotating drum.

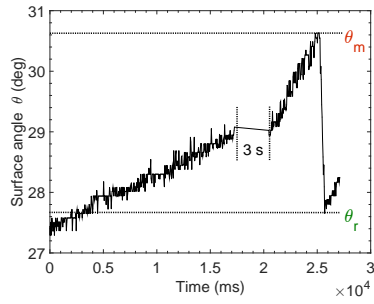
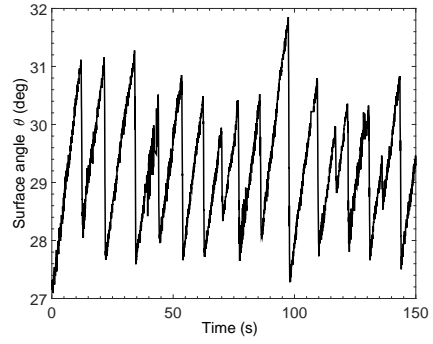


FIGURE 4: Top : Example of pre-measurement in the avalanche regime. This test enables to identify the destabilization regime and estimate the upper and lower repose angles. Bottom : Measurement of  $\theta_m$  and  $\theta_r$  during one avalanche, for  $n = 0.05$  rpm and  $H = 10\%$  in a heap without fines.

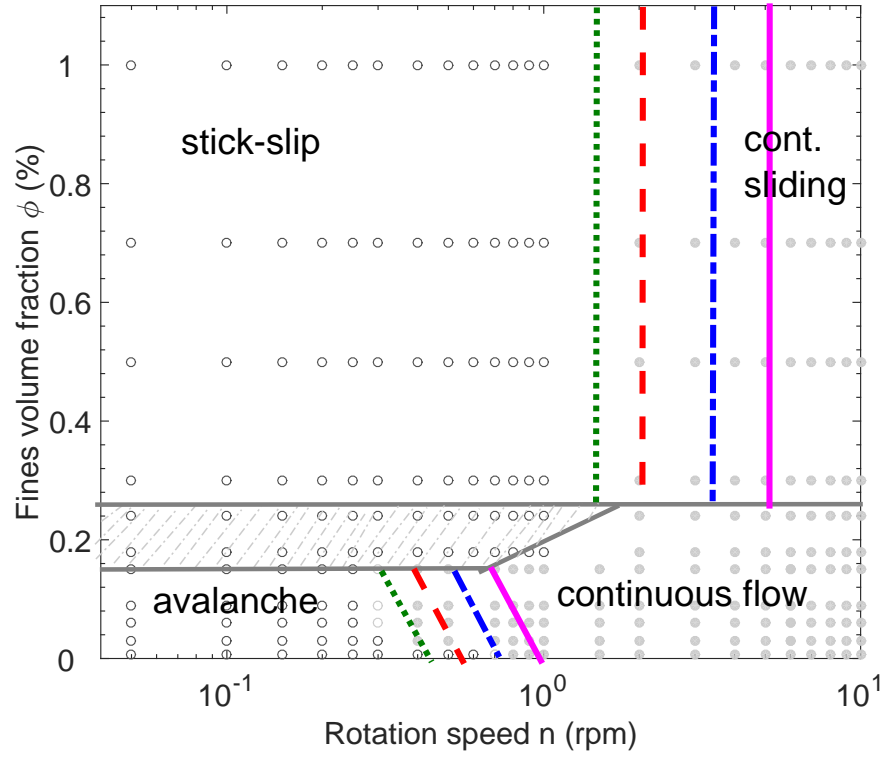


FIGURE 5: Diagram of stability of the granular pile as a function of  $\phi$  and  $n$ . Each dot stands for one experiment. Line segments share four different flowing regimes : avalanching, continuous flowing, stick-slip and continuous sliding. Between the avalanche and stick-slip regimes lies a small strip where both regimes coexist. Only the avalanche/continuous flow and stick-slip/continuous sliding separation lines depend on humidity, the others do not. From left to right, the relative humidity  $H$  is 10% (green dotted line), 25% (red dashed line), 40% (blue dashed-dotted line) and 53% (pink continuous line).

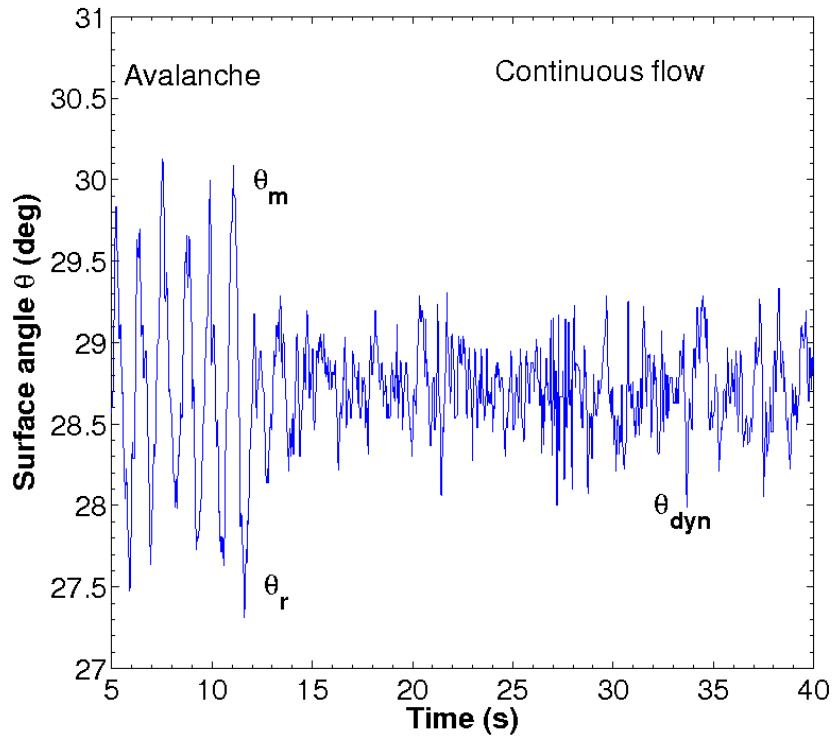


FIGURE 6: Angle of the surface of the pile as a function of time for 0.002% of fine particles, 25% relative humidity and 0.7 rpm rotation rate. Being in the intermittent regime, avalanche and continuous flow alternate during this experiment.

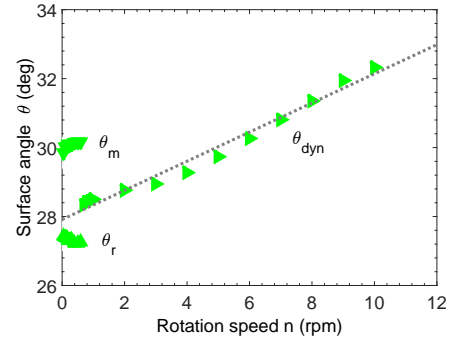
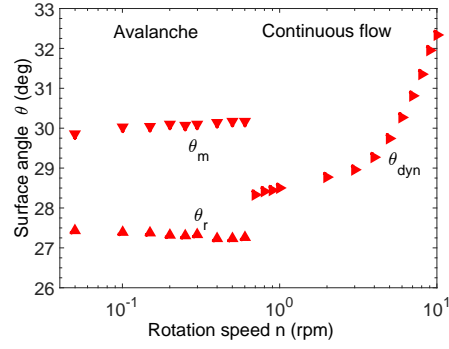


FIGURE 7: Avalanche angle, repose angle and dynamic repose angle versus drum rotation speed for  $H = 25\%$  and  $\phi = 0.005\%$ , in semi-logarithmic (top) and linear (bottom) scale.

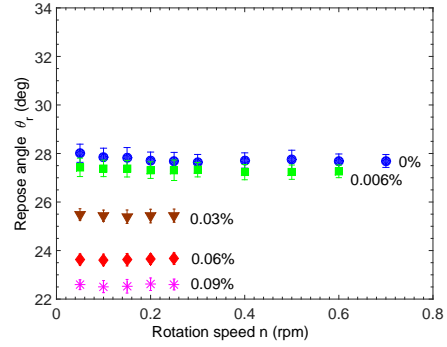
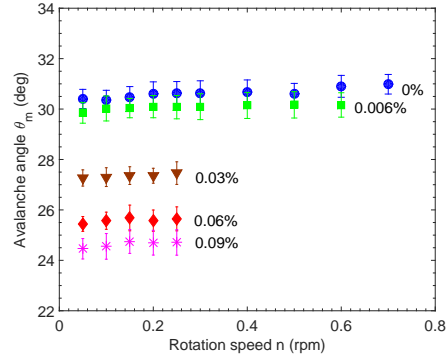


FIGURE 8: Avalanche angle and repose angle as a function of rotation speed for different fines volume fraction  $\phi$  for  $H = 10\%$ .



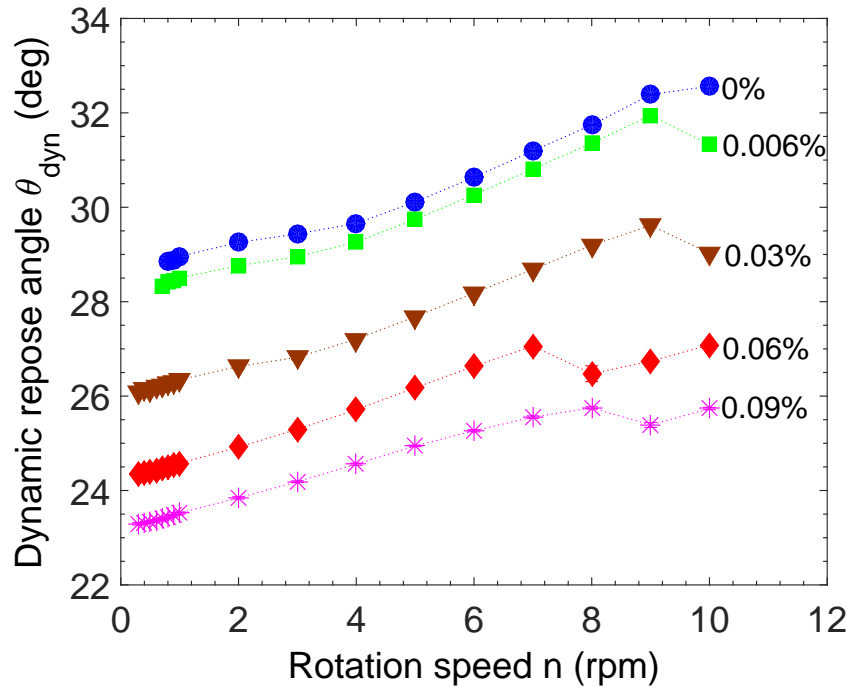


FIGURE 9: Dynamic repose angle as a function of rotation speed for different fines volume fraction  $\phi$  for  $H = 10\%$ .

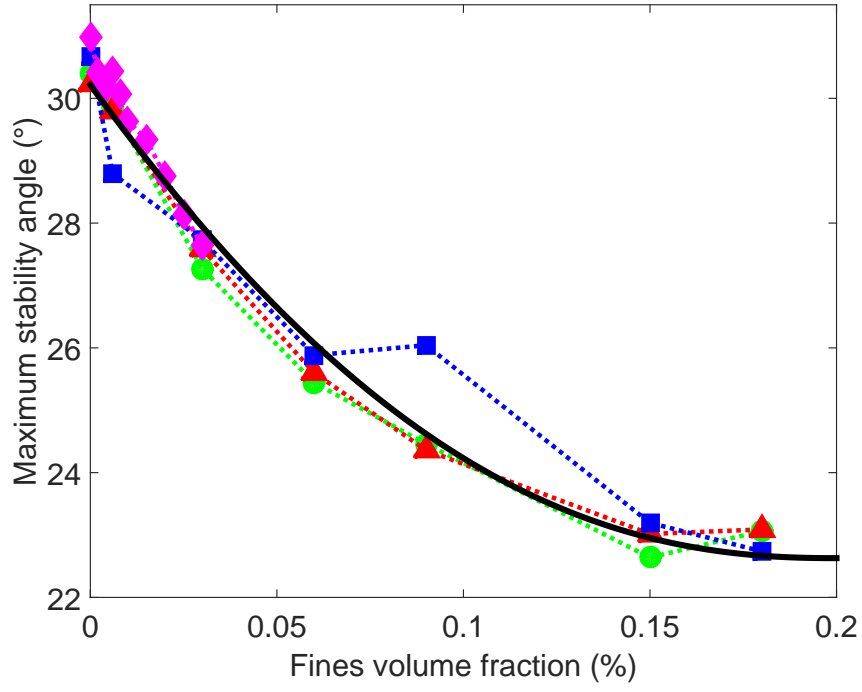


FIGURE 10: Maximum stability angle of the humid granular heap  $\theta_m$  vs percentage of fine particles in the avalanche regime ( $0 \leq \phi \leq 0.2\%$ ) for  $n = 0.05$  rpm. The color code for the relative humidity is the same as in figure 5. The black line is a fit of all the experimental dots by  $\tan \theta_m = \tan \theta_0 (1 + A(\phi_c - \phi)^n)$  with  $\theta_0 = 23^\circ$ ,  $A = 13.5$  and  $n = 2.3$  (see section 4.1).

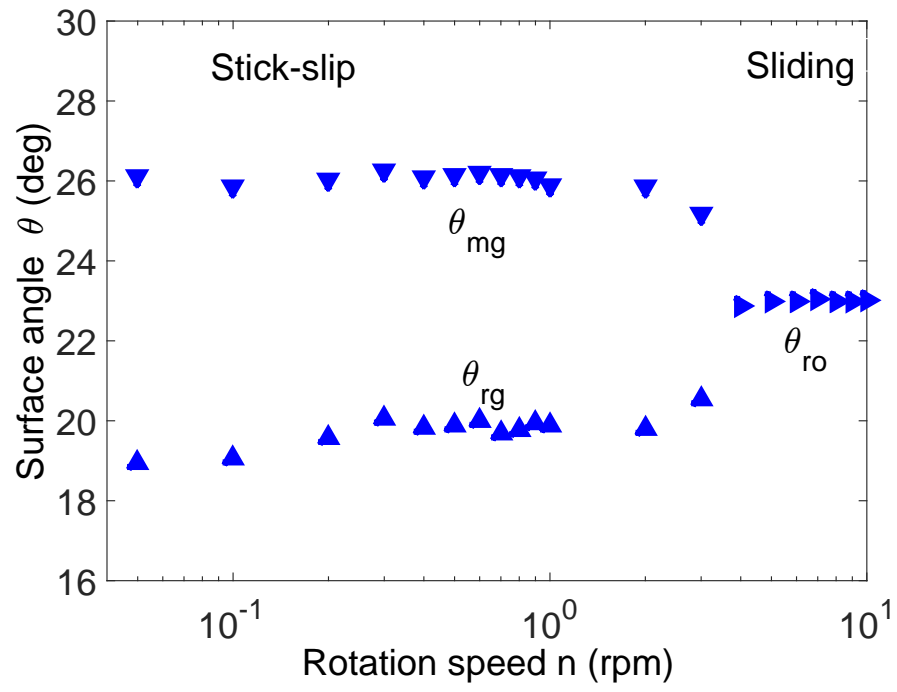


FIGURE 11: Upper and lower repose angles and sliding angle as a function of the drum rotation speed for  $H = 40\%$  and  $\phi = 0.5\%$ .

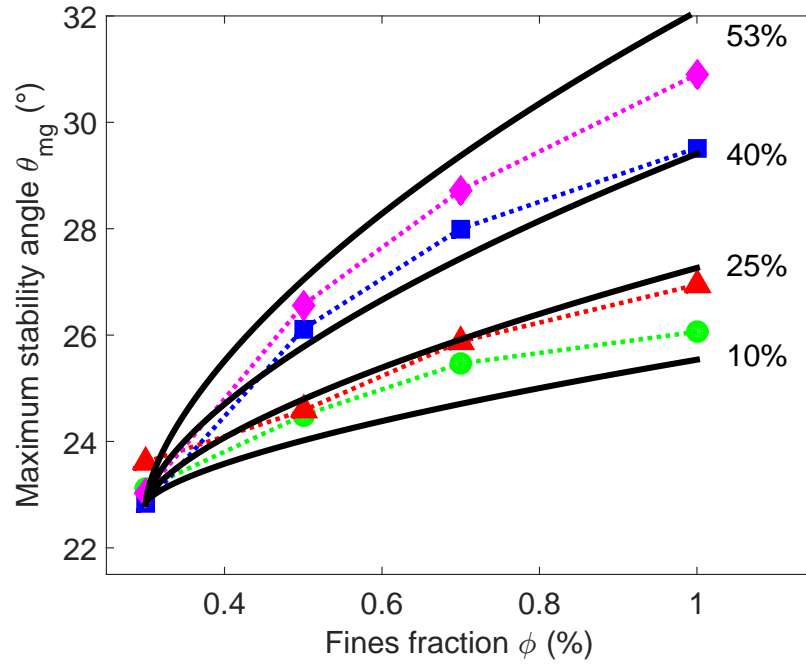


FIGURE 12: Evolution of the maximum angle of stability  $\theta_{mg}$  vs percentage of fine particles  $\phi$  in the stick-slip regime ( $0.2 \leq \phi \leq 1\%$ ) for  $n = 0.05$  rpm for various relative humidities  $H$ . The black lines are fitted curves of all the experimental dots using equation 3 with  $B = 3.3$  (see section 4.3).

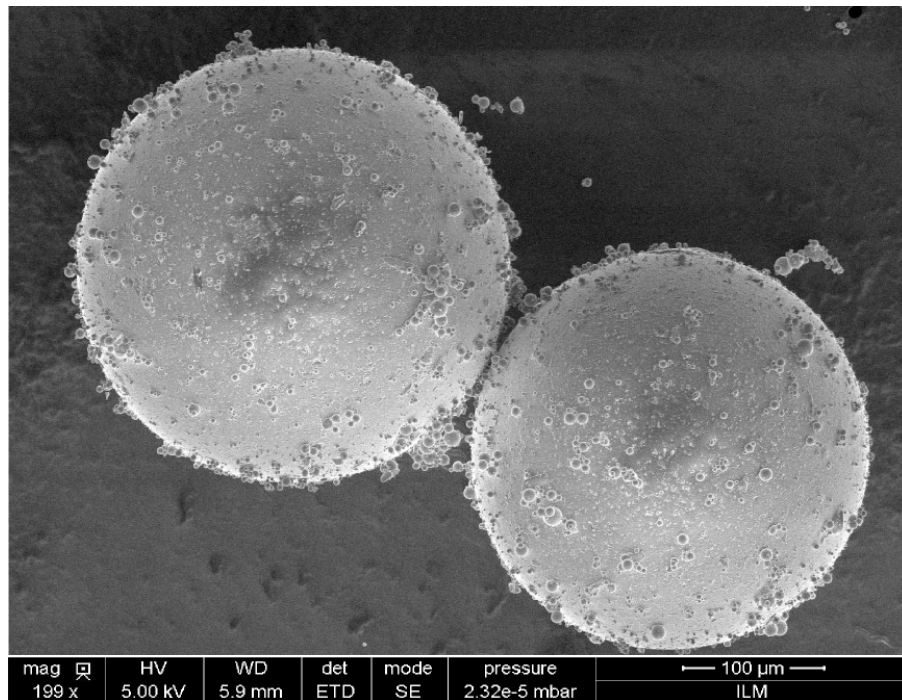


FIGURE 13: Scanning electron microscope picture of the distribution of fine beads between two large beads **sampled from a mixture** just after a rotating drum experiment **with  $\phi = 0.15\%$  and  $H = 40\%$** . As the experiments are performed in vacuum, the capillary bridges between the particles cannot be observed here, but we see that fines reconstruct a kind of meniscus between the large beads.