Flow of wet natural pozzolana

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Abstract. We present experimental results on the flow and stability conditions for natural pozzolana, a natural volcanic sand widely used in concrete production. We measured different angles involved in equilibrium conditions for sand piles and relate them to the flux parameters necessary to produce a silo evacuation. We vary some of the geometrical parameters in the silo to inspect the different flux responses of the system. Results are showed as a function of humidity present in the system. In this way, we related critical angles with flux conditions through a silo under different geometric setups and different humidity degrees, thus setting up a basic phase diagram for flux.

1. Introduction

A fundamental understanding of the behavior of real granular materials is crucial in many industrial applications. In order to address many processing and manipulating problems, researchers take advantage of experiments at the laboratory scale as well as of numerical simulations. A large number of studies have been done in this direction over the last decades, especially in the field of dry granular matter. In contrast, wet granular materials have received little attention and researchers very often try to avoid or minimize humidity both in experiments and models. However, several important applications involve, unavoidably, mechanical properties of wet granular media. Some examples include food processing, pharmaceutics, mine industry, geology and construction. Due to this strong relationship with applications, most of the research is aimed at providing empirical solutions to practical issues of wet granular systems [1-3].

Feng and collaborators performed interesting experiments in the field of packing of wet particles when capillary forces are dominant [4-7]. They measured porosity vs. moisture content in packings of spheres of different sizes, and derived a relationship between porosity and moisture content for both mono- and polydisperse packings of spheres.

They showed that the packing fraction (porosity) is strongly affected by the presence of moisture and observed three different regimes: a wetting regime where porosity increases with moisture content; a filling regime where porosity does not change with the addition of liquid; and a sediment regime in which porosity decreases with moisture content.

The interstitial liquid impacts on different physical properties of granular media, such us the angle of repose, which is the characteristic angle exhibited by a granular slope after an avalanche. Experiments reported by Hornbaker *et al.* [8] demonstrated that even a nanometer scale coating of liquid on millimeter-size grains can result in large changes of the angle of repose. In addition to the capillary force, ''wet'' particles experience a viscous force resisting motion, which can be derived from lubrication theory [9,10].

Among many examples, wet sand is one of the model systems for studying the behavior of moist grains. Our model system here will be a sand-like with substantial impact on cement production: the pozzolana.

Pozzolana, also known as pozzolanic ash, is a fine, sand-like volcanic ash (grain size less than 0.2mm), which owes its name to the city of Pozzuoli, Italy, where it was originally discovered. Finely ground and mixed with lime it creates a hydraulic cement and can be used to make a strong mortar that will also set under water. It transformed the possibilities for making concrete structures. These properties have encouraged its ubiquitous use in industrial processes, resulting in many manipulation problems.

It is common industrial practice to store raw granular material in piles before being used. This implies several issues that may be complicated, such as dead volumes due to repose angle; lost of material if it is dry; wetting of the material if it is stored outside; volume expansion due to moisture, and many more. On the other hand, a wet granular material is likely to get jammed when it is placed inside a chute to feed some mixing process. Thus, it is crucial to study the possible humidity dependence of the parameters involved in these manipulation stages.

2. Experimental methods and results

2.1. Density of a packing: wet dependence

The compaction capacity of a granular system is crucial for its flowing properties. Therefore, we first determined the dependence of the apparent density of the material on its humidity degree. We used an experimental technique similar to that in [4]. First, we pour a known amount of water in a container with Pozzolana sand. We then turn the container approximately 20 times to obtain a homogeneous mixing between the grains and the liquid. The mixture is then carefully poured through a funnel into a calibrated test tube. The resultant packing is weighted before and after drying it in a stove at 80 °C during two or three days. The water content W is determined as the percentage weight ratio between water and dry particles. We performed ten measurements (removing each time the used grains) under identical conditions in order to average the properties of the different packings (we work here with "loose" packings). The approximate W values investigated were: 0, 0.64, 0.68 and 1%. The behavior found for the apparent density is shown in figure 1.

We can see that the apparent density decreases with the percent of water present in the sample, from its initial value corresponding to a dry sample (approximately 0.74).

Due to the chemical composition of pozzolana we do not expect the grains to expand due to water absorption. Therefore, we can conclude that the expansion observed under the presence of humidity must be due to the presence of liquid bridges between sand grains.

The results presented in figure 1 can be also expressed in terms of the porosity of the system. The results obtained in this way show a good agreement with those found in pioneering studies, such as those reported in [4] for $W < 2$ %, or in the work by Yu *et al.* [11] and Zou *et al.* [7]. This discussion will be presented elsewhere.

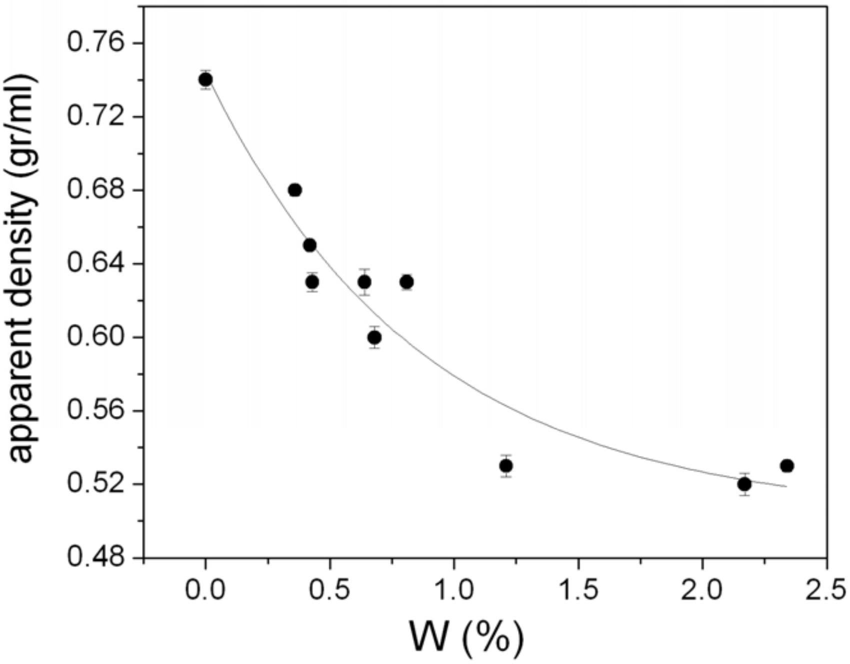

Figure 1. Apparent density as a function of the percentage of liquid content. The line is only to guide the eye.

2.2. *Stability angles for pozzolanic piles*

2.2.1. *Experimental set up.* Our objective in this part is to measure different critical angles related to the storing stage. We performed different kind of experiments based on the observed fact that the behavior of the banks of wet sand strongly depends on the history of formation of the pile. Thus, the measured angles depend on many details of the experimental set-up and the preparation procedure.

The experimental set-up consists of a glass box (25 × 45 × 30 cm). One of the vertical walls of the box can be removed and serves as a sliding gate (See figure 2). The height of the packing was chosen to be large enough so that the bottom wall has no significant influence on the stability of the pile. The box is attached to a rotation axis and a goniometer allows us to measure the angles with a precision of 0.5°. The granular packings are prepared by pouring the grains and filling the box up to the level of the lateral wall, as sketched in figure 2(b). All the measurements were performed with the same pozzolanic sample but at different wetting degrees. Humidity was controlled by drying the whole sample before a new experiment at a given humidity degree was started. Once the sample is completely dry, water was added in order to obtain the desired water-weight fraction. Note that this percentage is what we call humidity degree or liquid content. The added water is sprinkled over the grains and mixed in order to get a homogeneous sample.

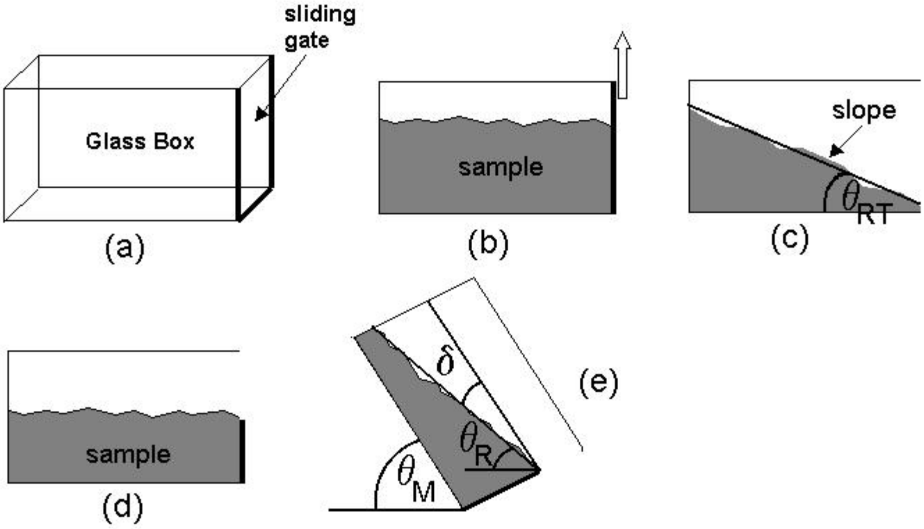

Figure 2. Set-up used for measuring critical angles. (a) Glass box used for the determination of all angles; (b) Front view of the box with a sample inside it; the right wall is being pulled upwards. (c) After the avalanche, the system relaxes to an angle θ_{RT}. (d) Front view of the box with upper half of the right wall removed (bold line). This set up is used to determine θ_R and θ_M. (e) After continuously tilting the wall, an avalanche occurs and rotation is stopped. Angles θ_R and θ_M are measured as indicated.

2.2.2. *Results.* The different critical angles as well as the experimental procedure used to measure them are described below.

Repose angle produced by sliding gate (θ_{RT}): a sample of the order of 0.1 m3 of volume is placed inside the glass box sketched in figure 2(a). The container is carefully set horizontal. The sliding gate is then quickly moved upwards and part of the grains flow out of the system spontaneously (figure 2(b)). A straight line that follows the surface of the grains left inside the box is then plotted on the glass wall (figure 2(c)). The box is then tilted until this line becomes horizontal which allows reading directly the value of the angle of repose θ_{RT} on the goniometer. We repeated similar measurements of for different filling heights of the glass box and no significant differences were found at the laboratory scale we were working with.

Repose angle by avalanche (θ_R) and maximum angle of stability (θ_M): The granular packing is prepared by completely filling up the box with grains, as depicted in figure 2(d). In this way, the height of the packing is determined by the height of the sliding wall. The box is initially horizontal and then carefully tilted at a constant rotation rate until an avalanche occurs (figure 2(e)). We define "avalanche" as the first sudden, simultaneous motion of several grain layers. The rotation is then stopped and the tilt angle of the container with respect to the horizontal is recorded. We also measure the slope of the surface after the avalanche stops and a new equilibrium has been reached (figure 2(e)). When the avalanche is triggered, the displaced material is always free to leave the container. Therefore, the slope of the surface after the avalanche corresponds to the angle of repose θ_R, and the tilt angle of the container is the maximum angle of stability θ_M.

Repose angle in conical pile (θ_C): this angle is the one related to the formation of a pile by pouring material from above at a constant, slow rate. The volume of the pile was of the order of 0.05 m^3. We measured the inclination of the conical surface at different positions over the surface, using an inclinometer, as sketched in figure 3. The average over these measurements determines θ_C. The values obtained in this way were compared with those measured over bigger heaps made at the industrial plant. No important scale effects were found on the results. This encourages further measurements at a laboratory scale.

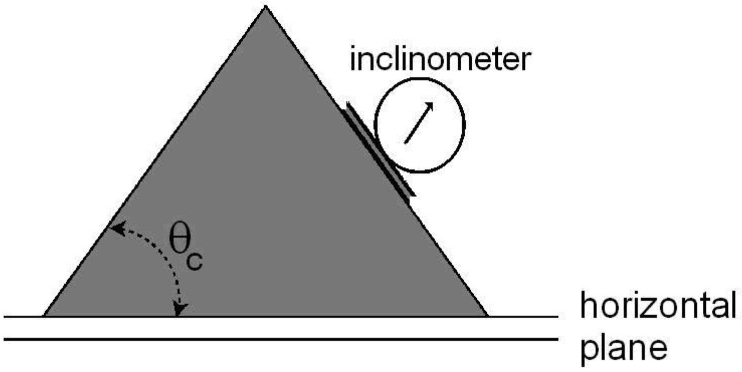

Figure 3. Sketch of a conical pile of pozzolana. An inclinometer was placed at different positions over the surface. θ_C and a position used for the inclinometer are indicated.

Figure 4 shows the variation of the characteristic angles as a function of the humidity degree. It is clear that the general trend of the stability angles is to increase with liquid content. The solid curves shown in the figure are guides to observe the tendencies. In experiments performed with dry material we observed that the grains were highly sliding and unstable. The measured angles in dry materials are systematically smaller than the corresponding ones for wet materials.

An interesting aspect is that the pozzolana system has "memory" with respect to the form in which the initial packing is prepared. Evidence of this is the large difference found between the values of the different characteristic angles θ_{RT}, θ_C and θ_R. This means that the chains of forces formed by grains in the initial packing (before inducing any type of avalanche) tend to maintain their network structure. These chains are reinforced by the presence of capillary forces due to inter-grain liquid in the sample. In addition, a significant increase in volume is observed as the humidity is increased. In figure 4 we see that θ_{RT} is systematically larger than the other angles, whereas the angle of rest in conical piles is always the smallest of the characteristic angles.

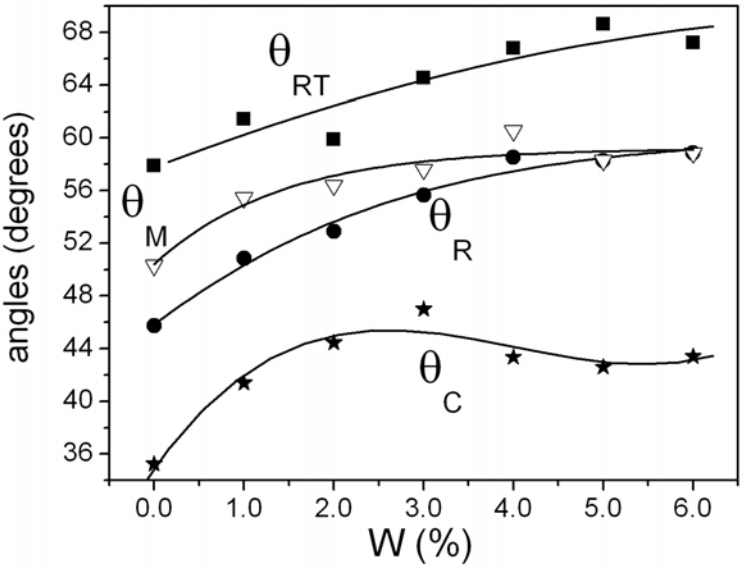

Figure 4. Stability angles for pozzolana sand as a function of wetting degree.

The behavior found for the characteristic angles is related to the method in which each avalanche is generated and, consequently, it depends on the effect of the chains of forces in the piling up. For that reason, they have different values. In the measurements of θ_{RT}, pozzolana is initially horizontal in the container, and the avalanche takes place due to the sudden rise of one of the sidewalls. Therefore, the effect on the internal chains is partial, and they are able to provide greater stability to the piling up. On the other hand, the measurement of θ_R involves a continuous rotation of the entire sample until an avalanche occurs. Therefore, the probability of breaking internal chains of forces is larger than in the previous case. Finally, the instability caused by the top flow of the material during the formation of a conical pile explains the smaller values of θ_C with respect to the previous angles.

Another important observation is that, as humidity degree increases, the evacuation of the material during the avalanche occurs in “blocks” of grains and superficial movements (so called “precursory” avalanches) are not observed. As humidity increases further, the values for θ_R and θ_M are practically the same, that is, the free surface is parallel to the base of the glass box. In this stage, the system behaves as a soft solid and the avalanche happens as a sliding block. This behavior (called Rankin effect) indicates that the characterization of the system as a granular material is no longer valid. For that reason, humidity values greater than 6% were not explored. Finally, it was observed that θ_R is the angle of stability that better characterizes the piles and it is the one showing the smaller fluctuations. In terms of its behavior as a function of humidity, this angle seems to reach a saturation value at which the solid-like features appear.

2.3. Flow in a silo

In order to determine the flow capacity of the material and relate it with stability conditions, we performed flow experiments in a cylindrical silo made of a zinc sheet, with diameter 33cm and height 100cm.

The exit consisted of a funnel that can be changed to vary the size of the outlet diameter. The diameter of the different outlets used in the experiments was: 11, 9, 7, 6, 5, 4, and 3 cm. The interchangeable funnels have the same height, h=21cm. Therefore, the angle α of the walls of the funnel can also be varied and is directly related to the outlet size of the hopper, i.e., to change the size the outlet means in our case to change the angle α of the cone of the funnel. A sketch of the silo and the interchangeable hoppers is shown in figure 5.

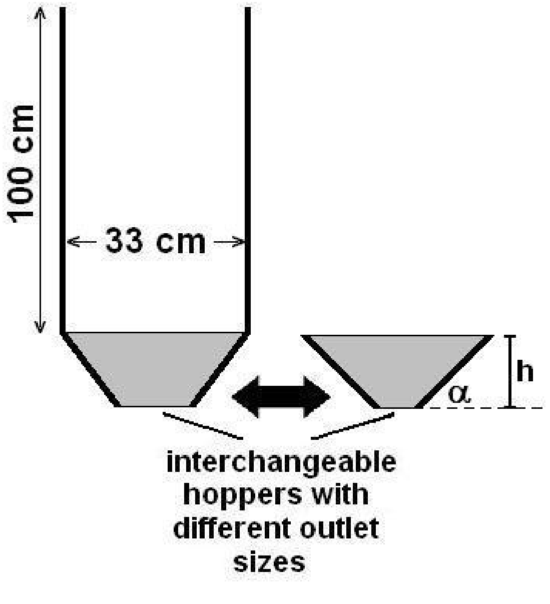

Figure 5. Sketch of the laboratory scaled silo used for the flux experiments. Interchangeable funnels were used with different outlet sizes. The height *h* is always the same and equal to 21cm and, thus, the angle α can be varied.

To have a better idea about the relationship between outlet sizes and angles of inclination of the funnels we present their corresponding values in the following table:

Table 1. Correspondence between angles and outlet sizes

Outlet Sizes (cm)	Angle α (°)
11	74.7
9	69.7
7	65.1
6	62.9
5	60.8
4	58.7
3	56.8

The silo, with its outlet closed, was filled with a weighted mass of pozzolana (ranging between 12 and 13 kilograms, approximately). The wetting degree of the material, W, is varied in order to study its effect on the flowing behavior. After the silo is full we open the outlet and the material unloads. The total time taken to vacate the silo is recorded. The average flow rate is then calculated through the ratio between the mass and the evacuation time. For each combination of W and outlet size, we repeated the experiment 10 times in order to average the results. It is important to mention here that the manipulation of the material induces the evaporation of some of the water added to it. For this reason we took several small samples during the different experiments in order to check the humidity values. Although we found low variation in these measurements, the wetting values W reported here are always an average over several samples.

During the discharge, the flow could eventually be interrupted. This jamming effect is due to the presence of arches created by grains during the flow process. This feature is enhanced by the presence of capillary forces in wet sands. When flow disruption occurred, we immediately tap the lateral walls of the silo to promote flow again. The procedure was so that the time employed during the tapping was small compared with the time for complete evacuation thus assuring an almost permanent flow in all the operation. The total time needed to complete the discharge (including taps) is computed as the evacuation time.

We classified the different flow features during discharge into two main sets: those presenting *jamming*, even if it only occurred once in all the ten experiments; and those *without jamming* where the flow was always continuous over the ten repetitions, i.e., no clogging was observed in the silo or at the outlet.

The inspected percentages of humidity, W, did not exceed 1%. Beyond this value, the evacuation of the material becomes impossible, even using the largest outlet of 11cm.

In figure 6 we show the diagram of different flow regimes, taking into account the wetting degree and the size of the outlet during discharge. As we pointed out before, two main zones can be distinguished in this figure, i.e., *jamming* and *no-jamming*. The lines on the figure separate these zones. Note, for example, that the point corresponding to an outlet angle $\alpha = 62.9^{\circ}$ and $W = 0.8\%$ (represented by a plus symbol) belongs to the jamming phase, while the one at $\alpha = 58.7^{\circ}$ and $W = 0\%$, represented by a square symbol, belongs to the no-jamming phase.

As the lines indicate, for angles larger than 60°, the jamming phase starts if the wetting percentage is above 0.35%. In fact, the left triangle drawn on figure 6, just below the line, represents a border case. During the experiments used to calculate this point, the flux was interrupted half the time by clogging. All points above the solid line have funnel angles greater than 60° or have humidity rate lower than 0.35%, so that they do not offer jamming problems for the flow through the silo.

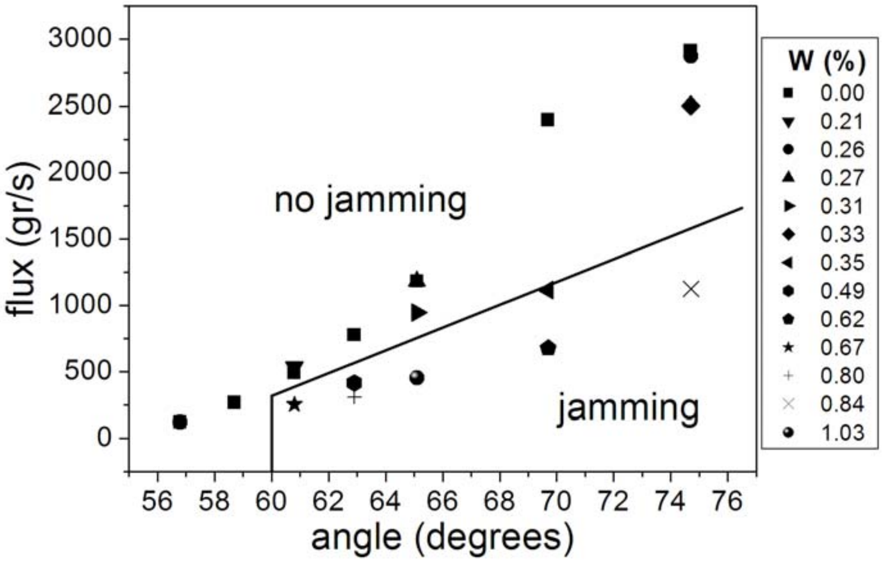

Figure 6. Phase diagram for the flux of pozzolana down a pilot silo. Jamming - no jamming zones are indicated. The angle indicated in the horizontal axis corresponds to the inclination of the walls of the exit funnel with respect to the horizontal.

It is useful to relate the results of figure 6 with those of figure 4 above. The angle θ_{RT} is the most relevant angle for the problem of flow down a hopper. This is the case because the experiments start from a static condition (full silo with its outlet closed) and move to a dynamic one triggered by a sudden perturbation (outlet opened). As explained in previous sections, the measurement of θ_{RT} is performed by suddenly removing the lateral wall of the container where the pozzolana was in a static condition. If we look at the values of θ_{RT} corresponding to a wetting degree between 0 to 1%, they are around 60º. This is in good agreement with our observations in flow experiments. Moreover, for W = 0%, figure 4 shows that θ_{RT} is close to 57º, which explains why all funnel outlets work fine for flow of dry pozzolana.

Finally, we observe in figure 6 that the flow rate increases non-linearly as the size of outlet in the funnel increases. This effect can be observed more clearly for W = 0% (squares), where we present the results for all the available outlet sizes. This may be related to the case where flow takes place through a channel formed within stagnant material and a funnel flow pattern will be developed. In that case, some material moves and the rest remains stationary during discharge from the silo. Funnel flow occurs when the sloping hopper walls of a silo are not steep enough and of the friction is not low enough for the material to flow along them [12]. Under these conditions, particles slide on themselves rather than on the hopper walls, and an internal flow channel appears. In this case, the flow rate depends on the hopper angle, on the size of particles and on the size of the outlet in a non-linearly way. The presence of humidity (typically > 10 %) does not meet the above pattern flow because the caking can occur when particles are stagnant.

3. Conclusions

The results presented here indicate that the stability angles as well as the flow of pozzolana have a strong dependence on humidity degree.

It was observed that dry material is highly sliding and unstable. All the measured angles are systematically smaller than the corresponding ones for wet material.

We also observed that the way in which a pile is prepared has a significant effect on the angles of stability. This "memory" effect is observed in the different values obtained for θ_{RT}, θ_{R}, θ_{M}, θ_{C}. In all cases, stability increases with wetting degree W.

On the other hand, the measurement of either of the stability angles, allows predicting the wetting degree of a given sample, knowing *a priori* a typical curve of behavior like anyone of those shown in figure 4. This is an important point for industrial manipulations in which there might be stages where humidity is present and hoppers must be designed adequately.

For values greater than 6% of humidity, the system behaves like a solid, making difficult its characterization by means of the stability angles.

Flow experiments conducted on the silo, using hoppers of variable outlet diameter, allowed us to establish the conditions of flow and clogging for pozzolana at different wetting degrees. The stability conditions (slope angle) of the material were related to the conditions for flow. Thus, we determined a simple phase diagram jamming/no-jamming that contributes to understand the necessary conditions for manipulating a wet granular material like pozzolana.

Finally, it is important to remark that given a particular real scenario, one has to choose which is the appropriate stability angle to characterize and predict the behavior of the system.

The results presented here are of direct application to larger scales and contribute to the basic knowledge of a very common system in cement industry, among others.

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