Development of innovative tools to characterize the drying of wet powders under shear

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1 Problem statement and project objectives

▷ **Granular matter and powders** are widely used in the manufacturing of numerous products and in many industries. In particular, powders are used as intermediate products but are also products consumed as such or, most often, after rehydration in the food industry [1]. They are also omnipresent in pharmaceutical products, but also in building materials as the PI has experienced with Saint-Gobain. Despite this intense utilization, their behavior is still poorly understood and empirical [2]. One significant difficulty in developing a general understanding is the range of possible powder properties (size, shape, wettability, etc.), the key role of the moisture and electrostatic force, and the flow conditions.

▷ **Drying of powders and formation of agglomerates.** Developing final products with powders often involves a wet agglomeration process, which is still very empirical [3]. Wet agglomeration of powders consists of coupling the agitation of solid particles to an operation of adding water or a binder to form granular structures of larger size, which after drying modify the properties of the final powder (flowability, rehydration properties, density, etc.) [4, 5]. At the industrial level, there is a great diversity of equipment (configuration, agitation mode) and modes of water supply (pulverization, flow...) allowing to realize this operation. The drying of wet powders ultimately leads to agglomerate formation because of solid bonds formed between the particles [3, 6]. The average size of the agglomerates produced can vary from ten to a few hundred microns. Nevertheless, in all cases, one can expect that two different drying techniques will lead to different final granules in terms of strength, size, compressibility, flowability, and ultimately result in products of varying quality and properties.

▷ **A complex (and impossible?) prediction.** As mentioned in the project brief, the question on how the intensity of the shear in a dryer affects the state of agglomeration of the dried product, and its redispersibility is broad and fascinating because of all the different physical and chemical ingredients involved. An approach to provide some first answers useful to a broad community would be to perform some real-scale experiments with a few selected powders and build a regime map for some well-chosen variables. However, this approach would have limited interest since the degree and nature of the agglomeration is influenced by the particle's surface chemistry and morphology (size and shape) but is also strongly influenced by the type of dryer used, as well as the presence or absence of solutes in the water. As a result, any particular characterization obtained may not be appropriate to describe or predict other configurations.

▷ **Should we give up? Towards a predictive tool.** The goal of this project is to develop two innovative experimental tools that will allow easy implementation and quick testing of a large variety of powders and liquid while controlling the input energy and/or shear rate during the drying process. We will base our approach on our expertise in granulation and blending of liquid and grains performed in the past with an industrial collaborator, Saint-Gobain. Deliverables will be the tools developed within this project from which we will obtain the final size distribution, but also the time evolution, of the agglomerates formed. The capabilities of such tools will be demonstrated through experiments with model powders, from which the PI will gain some fundamental insights into potential optimization properties (evolution of the final size distribution of the agglomerate with the shear rate).

▷ **Research objectives.** A schematic of the research objectives, tasks, and deliverables is shown in figure 1, highlighting the nature of the project and the cross-talk between the different stages. In particular, once the tools have been developed, the PI will seek powders of interest among the IFPRI members to leverage these characterizing tools while simultaneously running experiments with model materials. The proposed research will lead to the development of two characterizing tools, (i) oscillating box for high-shear rate and (ii) rotating drum for medium shear rate, that could be used on any powders to provide the time-varying and final size distribution of the agglomerates formed upon the drying of wet powders with shear. These tools will provide a first, quick, and cheap estimate of the influence of the different controlling parameters (shear rate, relative humidity, nature of the powders, etc.) prior to running more elaborate tests in industrial settings.

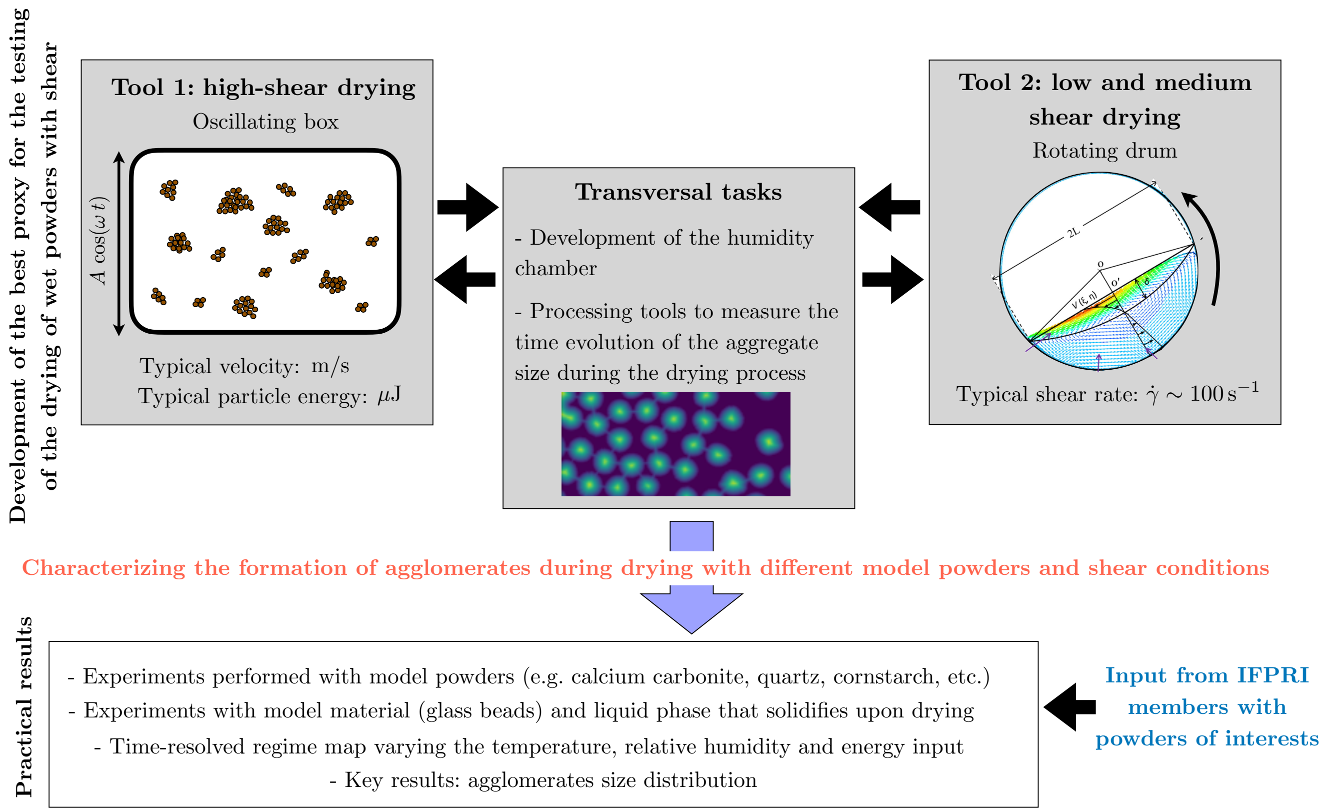


Figure 1: Schematic overview of the proposed research.

2 Expertise of the PI and relevance to its own research

▷ **Background of the PI.** The PI, Alban Sauret, is a faculty in the Department of Mechanical Engineering at UC Santa Barbara (USA) since 2018. He leads the Multiphase and Multiscale Flow Laboratory (*website*), which consists of 10-15 researchers. His group tackles problems in the area of fluid mechanics, soft matter, and granular materials. In particular, topics of current interest in the group include capillary flows of suspensions (dispensing of suspensions through nozzles, dip coating of suspensions, etc.), clogging in confined systems, rheology and properties of granular material and powders, blending of liquid and grains. Before joining UCSB, the PI was a CNRS Researcher between 2014 and 2018 in SVI (*website*), a joint academic-industrial laboratory between the CNRS and Saint-Gobain located in the Saint-Gobain Research center in Aubervilliers (France). In addition, he was a scientific consultant for Saint-Gobain Research in the field of granular materials, powders, and coating processes.

▷ **Why is PI well-qualified to develop new diagnostic tools for powders?** In the past, the PI, in collaboration with Saint-Gobain and Dr. Pierre Jop (SVI), has developed model approaches to gain some fundamental insights into the wet granulation processes. In these past projects, the goal was to work at constant water content and thus prevent any evaporation and drying of the liquid phase. However, similar approaches could easily be used and extended to control the drying of the liquid phase and provide relatively quick insights into the size distribution of the agglomerates resulting from the drying of wet powders. In addition, since 2019, the PI has started to investigate constitutive laws to provide a physical understanding of the concept of flowability of powders by studying the rheology of powders in various configurations in collaboration with researchers in France (Dr. Olivier Pouliquen and Prof. Maxime Nicolas, IUSTI, Marseille, France). Interestingly, whereas powders are handled at large scales, different simple tools have been developed to characterize powders in industrial environments (see for instance *Granutools*). The spirit of the present project is similar, *i.e.*, developing experimental tools to provide insights into the formation of agglomerates during the drying of wet powders under shear.

▷ **Why focusing on an oscillating box and a rotating drum as model configurations?** We have performed similar work leveraging the simplicity of an oscillating box in the past with a postdoctoral student and an MS student at CNRS and Saint-Gobain (France) to mimic the wet granulation process using controlled granular systems (spherical glass beads and polystyrene beads, both monodisperse in size). We thus believe that combining this approach with a control of the relative humidity, temperature, water content, and nature of the powders could lead to the development of an innovative tool for characterizing wet powders drying under controlled shear. Similarly, the PI has also considered the rotating drum configuration during the Ph.D. thesis of G. Saingier (funded by Saint-Gobain). We have used this approach to model wet granulation processes at low shear rates [7,8]. More specifically, we have considered the growth of a single wet aggregate rolling in a dry granular flow inside a rotating drum. We have been able to measure the time evolution of its diameter for different grains and liquids and various shear rates. Using X-ray tomography, we were also able to characterize the internal structure of the granular aggregate at different times during the process. Therefore, extending this approach to provide a tool to build a regime diagram of the drying of wet powders at low shear rates is feasible.

> The proposed project relies on the expertise of the PI and controlled flow configurations and drying kinetics. The goal is the development of innovative tools for characterizing the drying of wet powders with shear and the resulting formation of agglomerates. The tools that will be developed could be used with any powders and binding agents at high shear rates (oscillating box) and medium shear rates (rotating drum).

3 RESEARCH WORK-PLAN

Objective 1: Drying powders at high-shear

The first objective in this project will be to investigate and characterize the drying dynamics and the resulting agglomerates formed under *high-shear drying*, typically as encountered in flash and agitated dryers [9]. A large part of this process is controlled by the impact of the agglomerates with the impellers during the drying process [10]. Performing such an approach with an actual high-shear drying system would only result in the characterization of a specific situation (specific powder, for given relative humidity and temperature). However, these kinds of tests could be pretty time-expensive while only providing limited opportunities for optimizing the formation of the agglomerates. We, therefore, aim to develop a more controlled setup where we will be able to visualize the drying agglomerate both during the drying process using high-speed visualization and post-mortem (either with microscopy or X-ray tomography). During the drying process under agitation, the visualization of the agglomerates and the dynamic evolution of the size distribution may provide previous information on what controls the final size distribution (collision of agglomerates between themselves, on the solid boundaries, leading to the formation of larger agglomerates or break-up in smaller agglomerates).

▷ **From wet granulation to drying with shear.** Beyond the difference due to the drying phase, the wet granulation process [11] and the drying of wet powder under high shear rates share common key features. In the context of high shear wet granulation, the process often takes place in a tank in which rotating blades set the material in motion. The shape of the blades only impacts the intensity of the velocity field within the granulator, and they serve to set the material in motion and to give it enough energy for shocks to occur. Indeed, during the granulation stage, it is assumed that it is the shocks between agglomerates that will determine the average value of the final diameter of the latter [12]. It is, therefore, possible to assume that such an analogy can be made with the shear drying process. The approach is to use a large-amplitude vibrating pot where the two main control parameters to impose the shear are the amplitude of oscillation A and the frequency of oscillation $f = 2\pi/\omega$ [see figure 2(a)].

▷ **A simplified approach.** Although a vibrated box seems to be a simple system, in the context of wet granulation, the size distribution of the final agglomerate size with the water content have shown that the data obtained with this system are similar to data from high-shear granulation systems at an impeller rotation speed that would lead to a similar input energy in the system (close to 1 m/s with a typical size of the impeller of 10 cm and a typical particle size 10 μm) [13] although the details of the process are different. Moreover, it is worth mentioning that the effects of the typical velocity on the final size are also similar in industrial processes, suggesting that this setup was a promising model system to understand high-shear granulation, but also high-shear drying of powders [14].

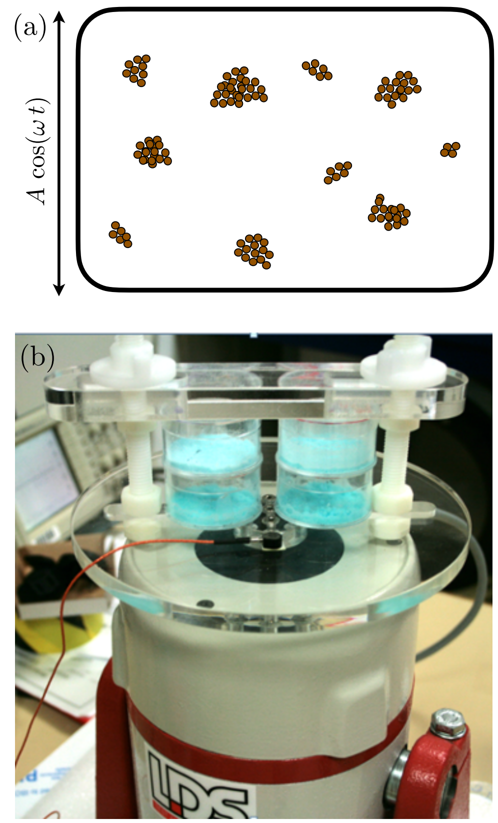


Figure 2: (a) Schematic and (b) picture of the oscillating box experiment.

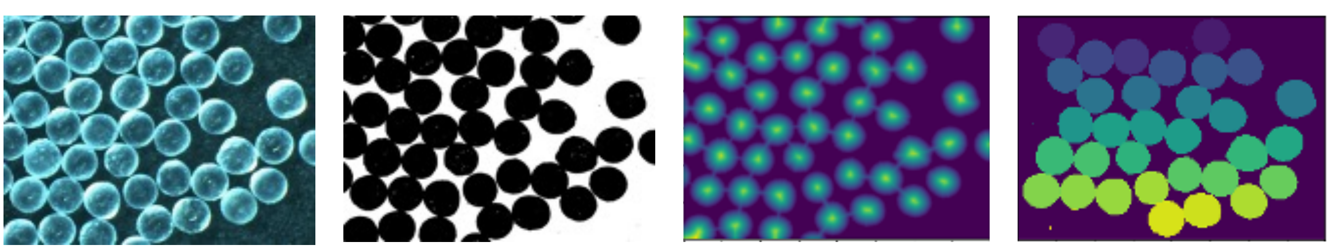
▷ **The apparatus.** We will initially base our approach on a setup similar to the one we used in the past, shown in figure 2(b). A small amount of the initial powder, mixed with the liquid, will be placed in a rectangular plastic box of typical dimensions $10 \times 3 \times 5$ cm. Within this box, shocks between agglomerates will also take place, similar to what would be observed in other high-shear drying processes. In addition, the agglomerates will also impact the solid boundaries during the entire drying process. This experiment should allow us to reach comparable values for the acceleration, velocity, and input with those obtained using a tank (the frequency and amplitude of the oscillations could be extended to reach a different range of shear). The mechanisms involved are likely similar, and this procedure seems to be a good alternative to provide a benchmark to test different powders, liquid, initial moisture, time variation, etc. To make this box oscillate, we will use a vibrating pot. The amplitude and frequency will be controlled by an amplifier and a low-frequency generator. We will also add an accelerometer on this plate to measure the acceleration experienced by the box, thus estimating the input energy in our system.

▷ **Preparation of the initial sample.** We will initially use a plastic bag in which we will weigh the desired quantity of powders, and the volume of liquid will then be added to obtain the desired liquid content with a pipette, and the bag will be sealed, the powder homogenized, and then place in the plastic box to run a test.

▷ **Environmental chamber: control of the temperature and the relative humidity.** An important modification to this setup will be to implement a control in temperature and relative humidity in the box. Since our system is quite simple, this will be easily done by connecting on the side of the box an inlet a full range humidity control with an elevated temperature that would allow control from 10 to 98% RH (at 20ºC) and a temperature from ambient to 55ºC. We have used such a system in the past, purchased from Electro-Tech Systems, within an environmental chamber, to study the drying dynamics in fibrous media [15]. The airflow can then be exchanged between this environmental chamber and the test box. An alternative will be to place the entire setup within the environmental chamber since its footprint is moderate. In both cases, we will have a total control over the temperature and the relative humidity. Furthermore, we would also be able to investigate the influence of the time-temperature history on the agglomerate.

▷ **Micro-High-Speed Imaging.** A specialty of our group is to characterize high-speed phenomena, such as, for instance, the formation of droplets of suspensions for manufacturing application [16, 17] or the blending of grains and liquids [18, 19]. These situations require reaching a recording speed of typically 10,000 frames per second and a spatial resolution of order 5 µm/pixels. We will use a similar approach here, where the motion and evolution of the agglomerate inside the oscillating box will be recorded using a Phantom VEO 710 high-speed camera (already available in our laboratory), equipped with a macro lens (Nikkor 200 mm), on which, if we need to reach a higher resolution, we could add a microscopic lens (Mitutoyo) as we have done in the past to study the coating of tubings by suspensions [20]. Even if high-speed imaging is not intended to record an entire drying under shear process, we will record a few seconds at different times along the process so that it will give us a picture of the entire drying dynamics, as well as crucial information on the state of the agglomerate at a given time. This approach is particularly unique, as it provides a direct visualization *in situ* of the dynamical process.

▷ **Image analysis.** For each experiment, the analysis of the size distribution of the agglomerates over time will be done using methods commonly used in our group (via custom-made routines). From the videos obtained with the high-speed camera during the dynamics drying process, we will detect the agglomerates as distinct ”objects” to obtain each equivalent diameter (defined through the surface area A by $D_{eq} = \sqrt{(4\,A/\pi)}$). The different steps are illustrated in figure 3 with an example for polystyrene beads [figure 3(a)]. The images of the agglomerates are first thresholded to differentiate the agglomerates from the background of the image [figure 3(b)]. Then a segmentation allows us to separate the agglomerates [figure 3(c)]. This step being done, the next step, the label, allows us to number these different objects to define each agglomerate as an object from which we can recover some characteristics, the diameter being the one we are interested in [figure 3(d)]. This method also has the advantage of following the fragmentation and coalescence processes of agglomerates and thus obtaining unique information on the dynamics during drying, such as, for instance, the time evolution of the agglomerate size distribution.



(a) (b) (c) (d)

Figure 3: (a) Example of a zoomed picture of agglomerate of glass beads and water formed during the model granulation process. (b) Example of resulting probability ditribution function of the agglomerate sizes and (c) Example of the evolution obtained when varying the size of the beads (blue: 60 µm, green: 200 µm)

Post-mortem characterization. Once the drying process is over, we expect to have a distribution of agglomerate that will depend on the particle's surface chemistry and morphology but also on the temperature, shear stress, and other physical parameters. We will be able to collect the samples and measure the relevant powder properties such as the bulk density and the flowability. In addition, we will image the resulting agglomerate using a Nikon Eclipse Microscope that will allow us to characterize more finely the final agglomerate size distribution as we have done for the granulation process, as illustrated in figure 4.

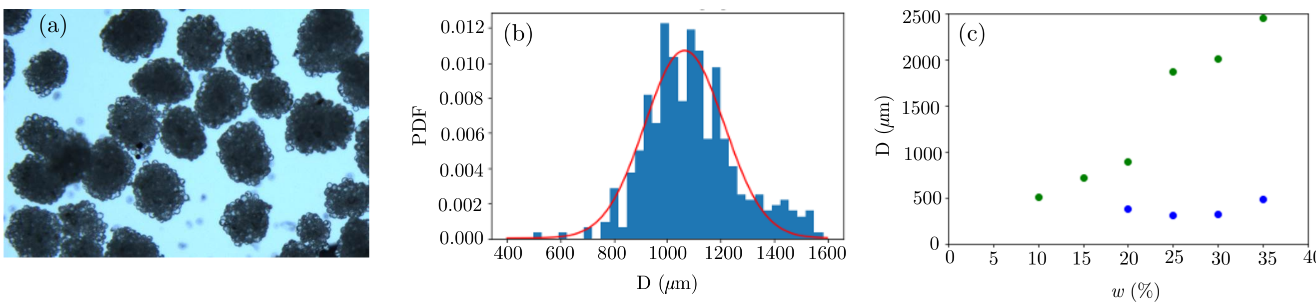


Figure 4: (a) Example of a zoomed picture of aggregate of glass beads and water formed during the model granulation process. (b) Example of resulting probability ditribution function of the aggregate sizes and (c) Example of the evolution obtained when varying the the water content w for two different size of beads (blue: 60 μm, green: 200 μm)

Expected outcome of objective 1. The development of the experimental setup and the characterization methods. The successful completion of this task will provide a unique tool to provide the final aggregate size distribution but also the evolution during the process to identify which steps may be the more important. This tool will then be used to characterize some powders and develop regime maps to identify the role of the time, temperature, relative humidity, and high shear stress on the drying of wet powders.

Objective 2. Drying at low and medium shear

Industrially, the processes of drying wet powders in the presence of shear to avoid agglomeration are diverse. The tool that will be developed in objective 1 aims at reaching high-shear and input energy, similar, for instance, to an agitated dryer. However, other methods, such as belt or rotation dryers, involve low or medium shear that will not be captured with the previous approach. The second main objective of the proposed project will rely on the development of a thin experimental drum, shown in figure 5(a), in which the shear will be imposed by the rotation rate, and the temperature and ambient humidity will be controlled in a similar approach while allowing in the same time to observe the drying dynamics.

▷ **Why is a rotating drum a good approach to model medium shear rates?** The rotating drum is one of the classical experimental configurations for the study of granular flows, which allows obtaining a stationary and cyclic flow. This system can also be used as a mixer [21] and has been shown to provide relevant information regarding the flow of powders [22]. In our study, the interest of the rotating drum lies in the periodicity of the flows that it generates, which makes it possible to observe the evolution of the agglomerates over long times, *i.e.*, during the entire drying process. The flows encountered in a rotating drum depend on the rotational speed of the cylinder, Ω. We will work in the continuous regime characterized by a stationary flow of grains at the surface. The flow of grains in the continuous regime occurs in two stages: (1) a rotation phase where the grains are at the bottom of the pile and have a solid rotational motion following the cylinder wall and then (2) an avalanche flow phase when the grains reach the surface. These two phases can be seen in figure 5(b). Models of the velocity field in a granular drum flow are well-known in the literature. The flow field exhibits a linear profile in the active layer (region II in figure 5(b)), which concentrates most of the shear, and a tail of exponentially zero-trending profile in the passive zone, having a solid-body rotation with the cylinder. The shear rate in the linear part is approximately constant and its amplitude is of the order of $\dot{\gamma} \simeq \sqrt{g/(4\,d)}$ [23], where d is the size of the particle or agglomerate. Since the velocity profile is linear in this region, the shear rate can be written as $\dot{\gamma} = V_{max}/h_0 = \bar{V}/(2h_0)$, where $\bar{V}$ is the average velocity in the layer and h_0 the thickness of the flowing layer that can be controlled with the rotation rate and the filling rate of the drum [24]. We will work in the situation where the thickness of the flowing layer is larger than the mean radius of the agglomerate so that it will be advected by the granular flow and subject to a controlled shear. We plan to perform the first tests in a range of rotational speed between 15 and 60 rpm. In summary, this configuration allows to quantitatively impose a rather low shear of the order of $\dot{\gamma} \sim 100\,\mathrm{s}^{-1}$.

▷ **Apparatus.** The experimental system that we have in our laboratory at UC Santa Barbara is illustrated in figure 5(a). This system consists of a cylinder with a diameter that can be varied (the first tests will be done with a diameter of about 10 cm, but this could be adjusted to provide more control over the shear rate). We will choose small drum thicknesses, typically of the order of the centimeter, to be able to measure in real-time the evolution of the roughness of the free surface, which ideally will give us information on the characteristic size of the agglomerates flowing at the surface. The cylinder is rotated by two rollers and driving at a rotation speed of 0 to 60 rotations per minute.

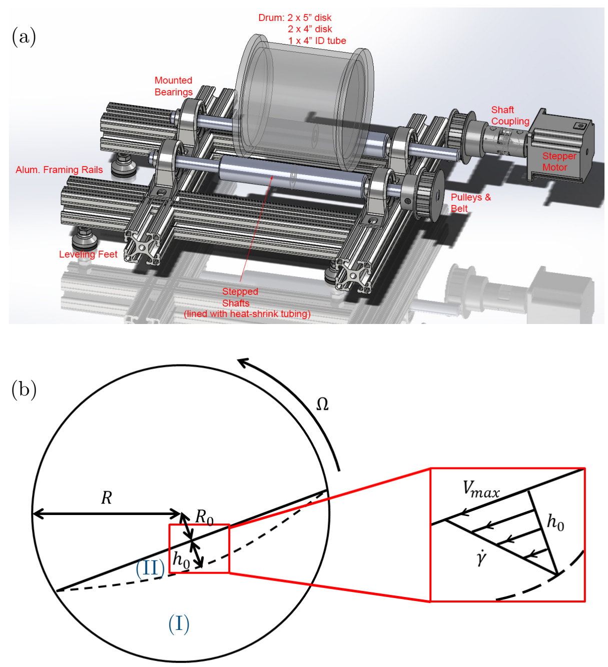


Figure 5: (a) Schematic of the rotating drum available in the PI's lab. (b) Schematic describing the properties of the flow in a rotating drum.

▷ **Principle of the measurements.** Similarly to the oscillating box described in the previous objective, we will prepare a sample of wet powders that will be placed in the rotating drum. The axis of the cylinders will be made of two holes so that the temperature and humidity will be controlled by placing the setup in the same environmental chamber. The experiments will start at $t = 0$, and we will visualize the flow within the rotating drum from the side. The roughness of the interface should provide us with information on the evolution of the agglomerates and, in particular, the cohesion between the grains. The rotating drum could easily be stopped during the experiments to pick up a small sample and measure the properties and size distributions of the agglomerate before resuming the experiments as we have done for the blending process in the past [8]. Methods similar to the one developed in the previous objective will be used.

Expected outcome of objective 2. The development of an experimental setup to characterize the drying of powders under medium shear and visualize the agglomerates during the process. The image analysis to obtain the time evolution of the particle size distribution will be similar to the methods developed in parallel to objective I.

Objective 3. Leveraging the tools: regime map of the resulting agglomerates formed

▷ **Validation of the methods.** The PI will use these two setups with model powders. The following ones have been identified as good candidates of hard inorganic powders: calcium aluminate, calcium carbonate, and silica glass sphere as they will not (or little) react with the liquid phase, *i.e.*, water within the framework of the project. Our goal is to choose materials that will not be too soluble to not precipitate when mixed with water. We will characterize the initial size distribution of each powder before using them in the experiments. Typically, we aim for powders of the size of order $10\,\mu m$ and initially, the liquid phase that will be used is water. We will consider the role of the following parameters on the size distribution of the agglomerates to build a regime map of the outcome of the agglomeration following drying of wet powders under shear: (i) shear rate $\dot{\gamma}$, (ii) initial water content, (iii) dynamics of drying (controlling the relative humidity and the temperature), (iv) size distribution of the initial powders. Of particular interest, especially with the oscillating box, is that we will be able to track the dynamic evolution of the size distribution.

▷ **Interactions with IFPRI.** The core of the project is the development of these two innovative tools and their testing on some model powders. After the first characterization made with the model powders, the PI will seek candidate materials (samples) from interested companies within the IFPRI consortium. In particular, it could be interesting to also qualitatively consider the configuration of soft inorganic materials.

Expected outcome of objective 3. The last objective of this project will be to demonstrate the relevance of the two diagnostic tools with model and more realistic powders. The main result will be the size distribution of the agglomerates under different drying dynamics and shear.

4 CONCLUSION

Outcome. The proposed project will develop innovative tools to characterize the resulting size distribution of agglomerates resulting from the drying of wet powders under controlled shear. The oscillating amplitude and frequency will control the input energy in the case of the oscillating box, whereas it will be controlled through the size and rotation rate of the container for the rotating drum experiments. In both experiments, the temperature and the relative humidity will be controlled thanks to an environmental chamber.

Tentative timeline. Year 1 will be devoted to the development of the oscillating box, the diagnostic methods, and model experiments with silica beads and water. Year 2 will be devoted to running experiments with more complex powders in the oscillating box (from which the PI will seek the input of the IFPRI members), as well as the development of the rotating drum experiments and its testing with the same model powders. Finally, year 3 will be used to build the regime map (dynamics and final size distribution of the agglomerate) for varying powders, shear rate, and temperature/humidity conditions. The key results will be the resulting average agglomerate size and dispersion of the distribution around the mean value. Similar to his past works, the PI will also look for theoretical approaches that could capture, at least qualitatively, these evolutions and could thus be used to provide some guidelines for industrial processes involving the drying of wet powders.

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