DRYING WET POWDERS WITH SHEAR TO PREVENT (*or control*) AGGLOMERATE FORMATION

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RESPONSES TO SOME COMMENTS FOLLOWING THE WINTER MEETING

1 - Key questions that were scheduled to be tackled in the first years of the project remain open.
The problem presented in the original brief is complex, as it involves modeling the fragmentation and coalescence of cohesive aggregates under shear and the drying process over a longer time scale. Our original approach, described in the response to the brief, was to develop a rotating drum setup and an oscillating box. During the first year of the project, we developed the oscillating box setup and obtained interesting results, but various members wanted us to reorient to an approach where the shear would be brought by air flows. Such an approach is also interesting to our group and led us to develop a third laboratory-scale setup, which was not initially planned. As a result, it took us a longer time to develop all the initial laboratory setups as initially planned, but we now have three apparatus that we aim to study further if the project were to be renewed.
Incidentally, a very close version of the oscillating box apparatus we worked on is now also used by other researchers (CNRS and Saint Gobain Research, France) to study the formation of aggregates of fibers [Link].

2 - The experimental platforms (apart from the rotating drum) produce shear fields that are too heterogeneous, making it difficult to extract a clear mechanistic link between applied shear and agglomerate behaviour.
The laboratory tools developed indeed have non-uniform shear fields, similar to any industrial system. However, we strongly believe that the final size distribution of the aggregate is controlled by the peak shear stress in the system. Indeed, the agglomerates recirculate in the system over a long period of time (the time for the powder to dry). Fragmentation events occur when the aggregates are subject to strong shear, and then, during the rest of their circulation in the system, they may coalesce again, but ultimately, we hypothesize that the aggregate size will be controlled by the peak shear stress. This peak shear stress will be localized between the two turbulent jets (for the air-jet setup) or when the grains are flowing on the top layer (in the rotating drum). Following some discussions at the last AGM, we took the time to develop model aggregates where we can control inter-particle cohesion with brittle bonds. Having these model aggregates now allows us to probe this hypothesis, as no coalescence will occur, and the system should converge to a final size distribution controlled by the peak shear stress if the hypothesis is true. In addition, such model aggregates could also be used in various projects (wetting of heterogeneous particles, grinding processes, etc.) as emphasized in our recent invited review article on experimental models of cohesive granular materials [Link].

3 - Is it possible to get more mechanistic understanding/theory and experimental setups that allow more precise shear application?
We agree that more model experiments allowing the development of frameworks to describe the

behavior of cohesive aggregates under shear are a crucial point in the community. We have actually been pushing in this direction, as shown by the proposal for renewal that has been developed with this idea in mind. For instance, Tasks 2.1 and 2.2 consider model situations to study the fragmentation at the aggregate scale. In addition to providing important information for the present project (since we can control the inter-particle adhesion and the shear environment and will be able to apply this knowledge in the laboratory tools), such results will also benefit future DEM models as a unique benchmark. The idea beyond Task 2.1 is to bring to the powder technology community the approach of atomization developed in the fluid mechanics community (*e.g.*, the work of N. Ashgriz on atomization). In addition, in this configuration, the structure of the turbulent jet is known, allowing us to provide a theoretical estimate of the shear rate applied. Similarly, Task 3 aims to bridge our microscale understanding of inter-particle adhesion with the macroscopic properties of cohesive aggregates. As discussed in the last AGM, it will be an important milestone for the community to develop *model aggregate* for which we know exactly at what shear rate they will break. Such aggregates could then be used in our large-scale system as *probe* for the shear exerted. In summary, we believe that the tasks that have been added to our initial approach proposed three years ago will allow us to bring a better mechanistic understanding of the behavior of cohesive aggregates under shear and will, for instance, allow us to estimate which flow velocity is required to break cohesive grains (after having measured their cohesive yield stress).

Proposed approach for the next three years

Granular materials and powders play a crucial role in industries such as food, pharmaceuticals, and construction, where they serve as intermediate or final products. The broad interest of academic and industrial research (including by members of IFPRI) for the past decades illustrates that understanding and predicting their general behavior remains a complex challenge. Indeed, industrial powders and grains exhibit a wide range of properties, including variations in size, shape, wettability, and sensitivity to moisture and electrostatic forces, all of which influence their flow and processing.

In recent years, our group has become increasingly interested in cohesive particulate flows and, in particular, in the behavior of particle agglomerates. Such groups of particles that are held together by capillary bridges or solid bonds are encountered, for instance, in industrial drying processes and wet agglomeration. The drying of powders and granular materials is a critical step in powder manufacturing, yet it remains largely empirical. Agglomerates are formed in industrial processes due to numerous factors, such as electrostatic attraction, capillary interaction between the particles, and moisture/binders, among others. Agglomerates impact properties key to the processing of bulk materials, such as flowability, rehydration, and density. In the case of drying, the output, *i.e.*, the agglomerate size distribution, and their density are highly sensitive to the drying technique, shear intensity, particle properties, and the presence of solutes. All these details often lead to variability in product quality and properties.

The first part of our IFPRI project has allowed us to initiate the development of laboratory-scale tools to study the effects of shear on drying wet powders. In the past two years, we have developed three tools to characterize and understand such processes at the laboratory scale. Such tools could be helpful to different types of powders (organic and inorganic) and liquids. In addition, we have identified several fundamental questions about powder technology that deserve further investigation through complementary studies. To build on this progress, we propose to pursue the development, testing, and validation of the experimental tools that would allow systematic testing of a wide variety of mixtures of powders and liquids under controlled energy input and shear conditions. These tools will enable the user to track the time evolution and final size distribution of agglomerates from a given initial composition, humidity, temperature, and shear applied.

1 Background: What we don't know and what (we think) we know

Agglomerate of particles: a sticky problem. When subject to a flow, both at small and large shear, wet particles and agglomerates formed through capillary cohesion are in constant collision. These collisions can lead to the merging of agglomerates (**coalescence**) or their breakdown into smaller agglomerates (**fragmentation**). As a result, at the end of the drying process, a distribution of the agglomerate size is obtained. Even having an order of magnitude of the final size distribution remains almost impossible at the moment. Yet, the first two years of the IFPRI project (2022-2025) have allowed us to narrow down some relevant questions and approaches that will improve our knowledge in this complex process and guide the development of laboratory tools that could, down the road, be used by different industrial partners.

Agglomerate of particles: between particle and bulk scales. As illustrated in our recent review [1], various experimental and numerical studies have focused on the behavior of cohesive materials, either in bulk scales or particle scales. The behavior and evolution of agglomerates have received considerably less general attention from the academic community. While industrial requirements have meant that agglomeration/deagglomeration processes of specific materials have received thorough investigation as required, a general picture remains elusive. One reason for this is that agglomerates are highly sensitive to the properties of grains, such as their shape, specific weight, magnitude of cohesion, etc. Agglomerate shape and size distributions are also sensitive to the processes and equipment that produce them, making a general framework quite challenging. In the case of cohesion due to capillary bridges, for instance, the methods of fluid introduction and removal can also substantially affect the final agglomerated result.

Shear: a challenging definition. In general, the application of deformation stresses to a bulk solid can cause it to break up into agglomerates when the magnitude of applied stresses is comparable to the cohesion in the system. These deformation stresses are sometimes called 'shear' stresses. However, in the dynamics of bulk solids in general, the presence of friction between grains means that any arbitrarily applied stresses have both shear and normal components. In the brief, we describe the use of shear stresses to mitigate agglomerate formation in the drying process. Following many exchanges with IFPRI members during the 2024 AGM, perhaps a better description is now using the term mechanical stresses in a general way since our approach is interested in the breakup of agglomerates using such deformation-inducing stresses. We should emphasize that the definition of 'shear' in the present context even varies between different IFPRI members.

Drying: from capillary bridges to solid bonds. Our project aims to investigate the drying of powders and granular material. Therefore, we need to consider two types of cohesion at the particle scale: (i) the cohesion that exists due to *capillary bridges* of water (or other liquids), and (ii) *solid bonds* that can arise from the drying of a liquid in which solutes are dispersed or due to the sintering of the particles during drying. We should emphasize that in an agglomerate, the two types of bonds can coexist during the drying process. The amount of liquid between the grains has a large effect on the cohesive force throughout the drying process. When drying powder using shear, the initial capillary bridges allow for the re-coalescence of aggregates, influencing the size distribution. When bonds are drying, the force between particles may increase. Thus, it may be desirable to continue breaking the capillary bonds to control the final size distribution. At the end of the drying, the bonds cannot be reformed but are often much harder to break. Therefore, there is a need to control and model the entire drying process.

What controls the final size of the aggregate? Even in the absence of drying, if one considers an assembly of cohesive particles forming agglomerates, it is unclear what controls the final size distribution in a system where the shear rate fluctuates spatially. Is it the peak shear stress (providing that all particles have time to pass through the location of peak shear stress a sufficient number of times), the average shear stress, or another similar quantity? In addition, during the drying process, the inter-particle force evolves, and one may thus expect a time-evolution of the agglomerate size distribution

2 RESEARCH OBJECTIVES

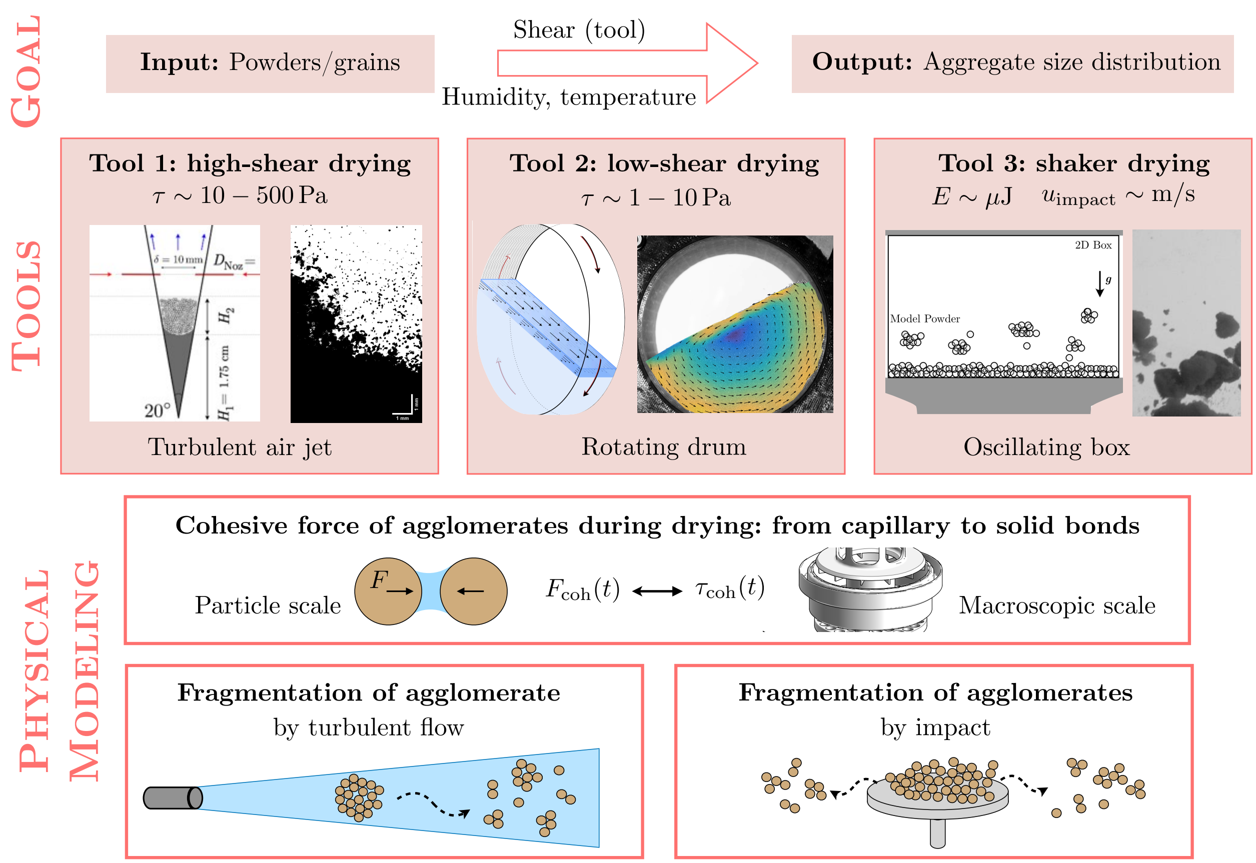
The first two years of the projects have allowed us to interact with different IFPRI members and get a sense of what each sees as issues in this complex and broad problem of drying under shear highlighted in the past brief. The physical situation is very rich and complex and could span years (or even decades of research). From our first experiments, results, observations, and discussions, we would like to pursue further this effort to address several questions:

- **Question 1.** *What controls the final size of aggregates exposed to a shear environment?* The different systems we have developed have all some level of shear and exhibit a peak shear at some physical location in the system. If the system is running for long enough, is the final size distribution and average agglomerate size controlled solely by the peak shear or by an average shear rate, or by something else?
- **Question 2.** *At the agglomerate size, what shear is required to break an agglomerate made of liquid or solid bonds?* To address question 1, an important point is to consider a controlled shear rate (imposed by turbulent jets or other methods) to understand and model the shear required to break an agglomerate (for a given size and inter-particle force) and predict the size distribution of the resulting aggregates.
- **Question 3.** *Can we provide a macroscopic yield stress for aggregates from the capillary scale measurements for solid bonds?* Rumpf's law expresses inter-particle capillary bonds to a macroscopic yield stress. Is a similar law valid during the drying process and, in particular, with solid bonds? Such a macroscopic description could simplify the prediction of the outcome of the drying as one could measure a macroscopic yield stress for a given powder both wet and after drying without shear.
- **Question 4.** *To estimate the outcome when drying a powder under shear, is it sufficient to compare the drying timescale and the residence timescale of the aggregate in the drying apparatus?* If yes, one could estimate experimentally and theoretically the timescale for drying agglomerates of different sizes with the timescale at which all particles would recirculate in the system.

The first two years of our project have allowed us to develop and characterize three experimental tools. We now want to extend this project to be able to first test model particles (glass beads mixed with a liquid that solidifies upon drying) and then representative industrial powders. Deliverables will be tools from which we could put a powder inside, set the desired shear (through turbulent jet, granular flow, or impacts), the temperature and humidity, and measure the final size distribution. Our discussion with different IFPRI members has also highlighted a need to provide some better modeling of the fragmentation process of cohesive aggregate, made either of capillary bridges or solid bonds. Finally, we will also monitor in real-time the evolution of the force between two particles during drying and try to build a framework from the particle scale to the bulk scale that could be used throughout different applications involving the formation of particle agglomerate due to the formation of solid bonds.

We will rely on approaches previously developed in our group that use model systems to emulate shear rates reminiscent of different shear dryer systems. Since some of those questions are also of fundamental interest (for instance, the output of fragmentation or coalescence of agglomerate), we will also be looking at additional academic funding, such as from the National Science Foundation, which would ultimately also benefit the powder technology community and IFPRI members.

The figure below shows a schematic of the research objectives and tasks for this three-year renewal project. It highlights the different tools developed and the different scientific questions that will help us rationalize the observations and results obtained with the tools.



3 PRIOR DEVELOPMENT - EXPERIMENTAL SETUPS

The first two years of the project have allowed us to develop different experimental setups, thanks to the input of IFPRI members. These experimental setups allow imposing tunable and controlled shear or input energy on wet and drying powders and grains. We now aim to renew the support from IFPRI to leverage these experimental tools to advance our understanding of the evolution of drying agglomerates under shear. One of the goals is to provide industrial partners with "turnkey" tools where humidity and temperature would be controlled and where the shear or energy inputs can be adjusted. The operators would then be able to use small powders or granular material samples with the corresponding liquid phase in these lab-scale tools to measure the final agglomerate size without needing to conduct large-scale industrial tests. This would serve industrial partners as a first simple measurement of what to expect and in which direction to tune the shear, temperature, or humidity.

To ensure the reliability and validity of these tools and the relevance of the output measurements, we also plan to study physical situations representative of part of the complex dynamics in the experimental tools. These side experiments will directly help interpret the results obtained with the innovative laboratory tools. Below, we briefly outline the principles and design of these setups. The following section will present the future developments we aim to work on pending a potential renewal of support from IFPRI.

High shear in air: impinging turbulent jets

The "high-shear" tool consists of a thin cell, allowing visualization during the process, and where two nozzles are facing each other, resulting in the creation of an intense velocity field and turbulent shear in the region between them. During the development of the tool, we quantified that, in the absence of particles, the shear exerted between the turbulent jet is of order $\tau_{\max} \approx [30\,\text{Pa}, 200\,\text{Pa}]$. Decreasing further the diameter of the nozzles (currently $D = 0.7\,\text{mm}$) will allow us to increase further the shear.

We are currently adding additional airflow to promote more circulation in the granular bed, although, at high intensity, most particles are already periodically subject to turbulent shear. One question we are currently looking at in Year 3 of the project is the amplitude of the shear in the presence of particles.

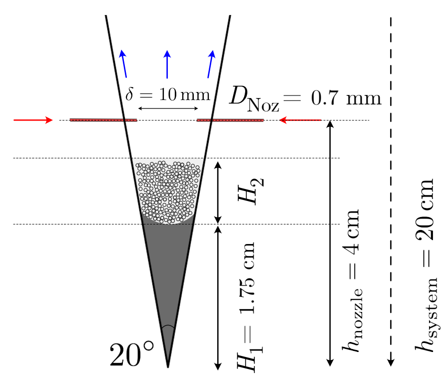


Figure 1: Schematic of the experimental setup used to break the aggregates with two impinging turbulent jets creating a local high shear.

Low shear in granular flow: rotating drum

The second laboratory tool we developed and characterized during the first years of the project is a rotating drum in which the drying of wet granular material is possible. Whereas some drying methods involve large shear induced by airflow, other methods, such as belt or rotation dryers, involve low or medium shear. The rotating drum that will be used is shown schematically in figure 2 (currently: diameter $D = 7.5\,\text{cm}$ and length $L = 10, 20\,\text{cm}$ rotating between 0.1 rpm to 20 rpm). One advantage of such a setup is the possibility of running experiments for hours while having air circulation with a controlled temperature and humidity through the cylinder's axis.

In this system, we can visualize the flow at the wall and estimate the aggregate size through the sidewall. However, the most interesting method to measure the time evolution of the aggregate size is to stop the rotating drum, pick up a small sample, and measure the size distributions of the agglomerate before resuming the experiments. Controlling the rotation rate and the filling of the drum allows tuning the shear rate. The characterization performed previously shows that we can have shear rate or order $\tau \sim 1 - 10\,\mathrm{Pa}$ (note that tuning the diameter of the rotating drum will allow us to explore a larger range).

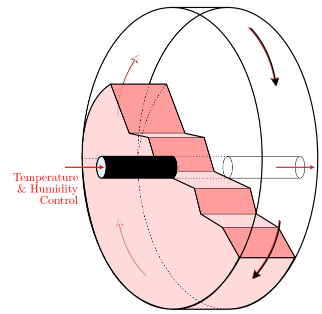


Figure 2: Design of the rotating drum with connection to temperature and humidity control.

***Collision driving break-up*: oscillating box**

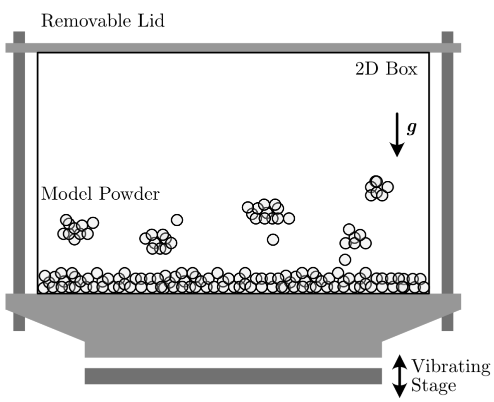


Figure 3: Schematic of the shaker: the apparatus is attached to the vibrating stage by a 3D-printed fixture. The lid can be removed, allowing the model powder to be fed in or removed.

We developed during the first year of the project an apparatus that provides mechanical stresses to the wet granular material through the impact of the agglomerates with the container walls or between themselves. The mechanical agitation is induced by the fast oscillation of the box placed on a mechanical shaker, as illustrated in figure 3. The inspiration for this apparatus came from Ref. [2], who aimed with a similar setup to mimic the impact of the agglomerates with the impellers [3].

The important input parameter is the acceleration of the box $a = A\,\omega^2$, where A is the amplitude of oscillation and ω is the oscillation frequency of the box. The wet powder is initially placed in the box. An LED light panel is placed on one side of the cell, and a high-speed camera is placed on the other to image the effects of the agitation on the agglomerates. We can then follow the time evolution of the aggregate size distribution but can also extract the outcome of agglomerate-agglomerate collision, which would help benchmarking and improving the numerical simulations of such processes.

4 Research work-plan

To address the questions raised in section 2 and finalize the testing and characterization of the tools developed, we will organize our efforts around the research tasks described below. Note that the tasks are not sequential and some aspects will be considered in parallel as the knowledge gained from some experiments will allow us refining our description of the laboratory tools.

Task 1. Time-evolution of dried agglomerates under shear

▷ **Task 1.1. Air jets tool.** We will first perform experiments with this tool in the absence of drying, *i.e.*, for aggregates connected by solid bonds for which we can measure the inter-particle force and the macroscopic cohesion. Together with the knowledge that will be gained in Task 2.1, we will be able to *determine if the peak shear controls the final size distribution of the agglomerate or if an average description is more relevant*. In addition, extracting samples at different locations in the setup at the end of the experiment will also help us to ensure that all the agglomerates are circulating in the system and thus pass at some points through the region of high localized shear. Since the threshold shear to break an aggregate of a given size will also be obtained through model experiments in Task 2.1, it will allow us to fine-tune the value of the peak shear in our system to be used in Task 3 where we will add the drying component.

▷ **Task 1.2. Rotating drum.** Similarly, in the rotating drum, the value of the shear changes with the location in the flowing layer. Using model aggregates for which we will know the inter-particle cohesion will also allow us to estimate the shear required to break up the aggregate down to a given size. These experiments will be particularly useful when then considering the role of drying.

▷ **Task 1.3. Oscillating box.** A similar experiment will be performed in the oscillating box, with one advantage: we will be able to track with the high-speed camera the time evolution of the size of the aggregate. Note that using solid bonds will also only allow fragmentation of aggregates and not coalescence, thus simplifying the estimation of the energy input and leading to the breakup of the agglomerate.[1]

> The experiments with model aggregate where the particles are connected with solid bonds of controlled cohesive force will allow us to estimate how small one may expect to decrease the average size of dried agglomerate. These results will be useful for comparison with the experiments where drying will occur. They may also be of interest to IFPRI members who may be left after some long-term storage of powders with bonded particles that they want to re-fragment into smaller agglomerates (for example, powders for the food industry exposed to humidity leading to the sintering of particles).

Task 2. Fragmentation of particle aggregates connected by liquid and solid bonds

The drying of agglomerate under shear is intrinsically multiscale (in time and space). A complex dynamics occur in the rotating drum (or for any flow where the shear is generated by flowing grains), or when the shear is exerted by a localized turbulent jet. Discussion with IFPRI members and academic partners, it appears that this complex situation opens fundamental and practical questions for powder technology: *many studies have been done these past 20 years to understand and predict the atomization of Newtonian and complex fluids [4], including from our group studying the suspension of slurries and droplets [5,6]. Yet, despite the technical improvements due to high-speed cameras, a similar understanding has not been developed for aggregates made of cohesive grains or connected through liquid/solid bridges.*

▷ **Task 2.1. Fragmentation of a cohesive aggregate by a turbulent jet.** A model cohesive aggregate will be formed by mixing together (i) glass beads and a Newtonian liquid binder, (ii) glass beads with solid bonds formed upon drying of an initially wet aggregate, and (iii) industrial powder such as calcium carbonate initially wetted and then dried to form an agglomerate. One may expect a different fragmentation process since capillary bridges need a threshold distance for the cohesive bond to break. The principle of the experiments we want to perform is shown in figure

[1] Our focus through the renewal of this project will remain mainly on the air jets and the rotating drum, and we may seek further funding from agencies to explore further the configuration of the oscillating box and the fate of agglomerates. Some exchanges have been initiated with a program manager of the NSF.

4. This approach is inspired by recent experiments on the atomization of liquid droplets that have improved the understanding of liquid atomization [7].

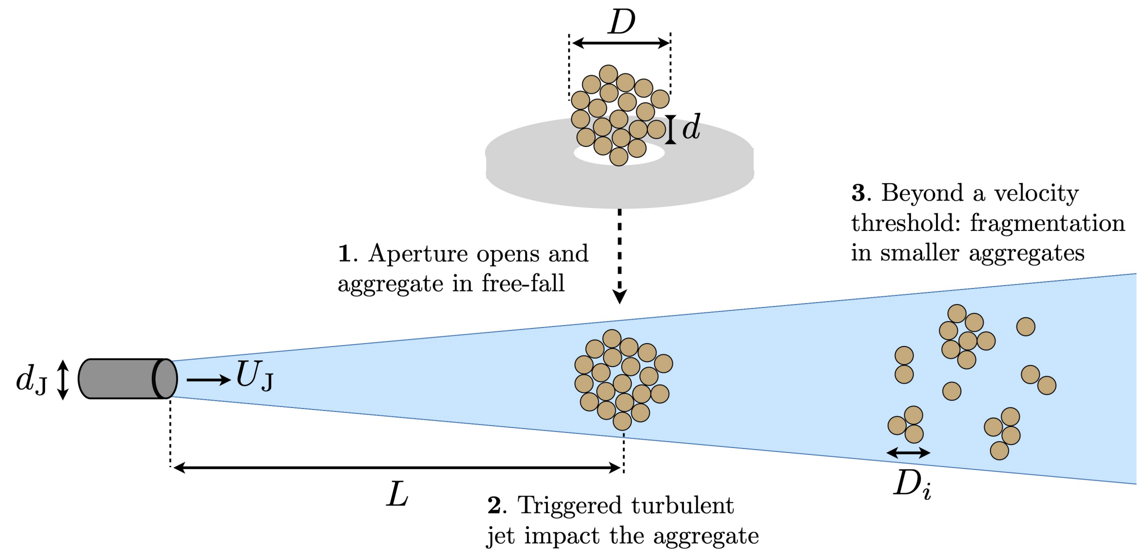


Figure 4: Experimental approach to investigate the fragmentation of a cohesive aggregate by an impinging turbulent jet.

At time $t = 0$, the aggregate is released from an opening (through a diaphragm that suddenly opens and lets the aggregate fall), and through a trigger system, a turbulent jet exiting an axisymmetric nozzle of diameter d_J, located at a distance L from the aggregate, suddenly blows a turbulent jet at an outlet velocity U_J. Our group has already been working on turbulent jets and is qualified to characterize all the associated hydrodynamics [8]. One can expect that below a threshold amplitude of the airflow, the aggregate will just be carried by the turbulent jet. Beyond this, it is unclear how the aggregate will break into multiple pieces. Yet, the prediction of the size distribution and the threshold will be of importance in modeling Task 1.1.

▷ *Control variables and output parameters*: As can be seen in figure 4, different parameters will control the output of the fragmentation process:
◦ **Turbulent jet:** diameter of the nozzle d_J, outlet velocity U_J, distance to the aggregate L.
◦ **Cohesive aggregate:** diameter of the particles d, of the aggregate D, inter-particle cohesion F_c.
The output quantities that will be measured through high-speed imaging are the size distribution of the fragmented aggregates D_i and the average size $\bar{D}_i$. One may expect to find a Gamma distribution or a log-normal distribution (the exact distribution will likely depend on the reversible or irreversible nature of the cohesive bonds). The average size $\bar{D}_i$ will depend on the aforementioned parameters.

▷ **Task 2.2. Fragmentation of a cohesive aggregate by impact.** The second aggregate-scale experiment we want to develop is inspired by the *"shaking box"* experiment proposed in the previous project (and itself inspired by blades impacting aggregates in a granulator). Although there has very recently been a numerical approach to the evolution of a cohesive aggregate impacting a substrate [9], experiments with more realistic parameters, which would allow the development of a framework to describe the fragmentation of agglomerate, are still missing. We will consider model and "industrial" aggregates as described in Task 2.1. The experimental setup we will rely on is shown schematically in figure 5.

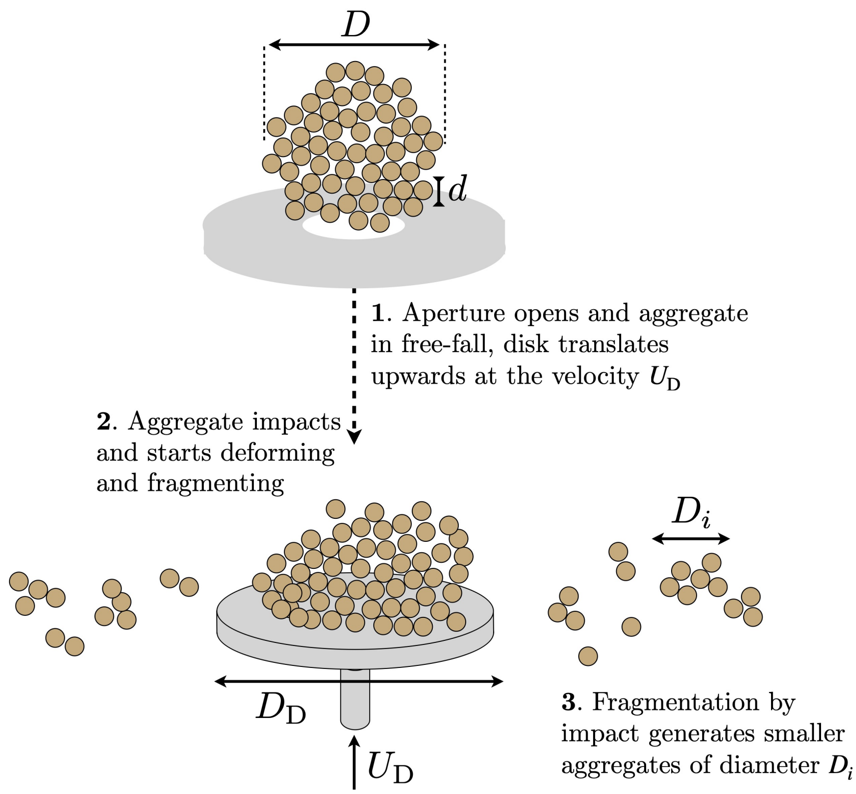


Figure 5: Experimental approach to investigate the fragmentation of a cohesive aggregate by the impact on a solid surface.

The release mechanism will be similar: at time $t = 0$, a diaphragm will open and let the aggregate of cohesive particles fall under the effect of gravity. The fall of the aggregate of diameter D, will trigger the upward motion of a solid target of diameter D_D at the velocity U_D (much larger than the falling velocity of the aggregate). The diameter D_D may have some influence on the outcome of the fragmentation, and both the situation $D_D \sim D$ and $D_D \gg D$ will be considered. Upon impact, if the energy brought by the impactor is sufficiently large, it will break the aggregate into smaller aggregates. The fast dynamics (a few milliseconds) will be recorded with high-speed cameras available in our group, and image analysis will lead to the size distribution of aggregates $p(D_i)$.

Similarly to the turbulent jets, various parameters are expected to control the output of the fragmentation process:
Impact of the disk: impact velocity U_D (the size of the disk D_D) may play a role, but this role is expected to be of second order).
Cohesive aggregate: diameter of the particles d, diameter of the aggregate D, inter-particle cohesion F_c.

Again, we will obtain the size distribution of the fragmented aggregates $p(D_i)$ and aim to develop some general scaling law. One may expect that the energy of impact $m_{agg}\, {U_D}^2$ will be a key parameter.

The deliverables of Task 2 will be a classification of the different possible fragmentation scenarios (likely (i) no fragmentation, (ii) partial breakup, and (iii) total fragmentation), the identification of the mathematical distribution capturing the size distribution of aggregate, and the average aggregate size after fragmentation. The results of this task will provide information on the minimum velocity of the turbulent jet to use in the tools described in Task 1.1. The results on the impact of agglomerate will also help modeling the tool developed in Task 1.3.

Task 3. From the particle scale to the macroscopic scale

As emphasized in our recent review on cohesive granular material (available in the Year 2 report), connecting the particle-scale cohesion to macroscopic yield stress would allow the development of better scaling laws for drying particles under shear and, overall, of many processes involving cohesive particles.

Evolution of the inter-particle force during drying. We have developed a method to measure the force between two particles connected by a liquid bridge during the drying of the liquid bridge while also imaging the formation of the solid bond [figure 6(a)]. An example of the result of the system is shown in figure 6(b), where we observe that the amplitude of the force increases strongly during the drying process.

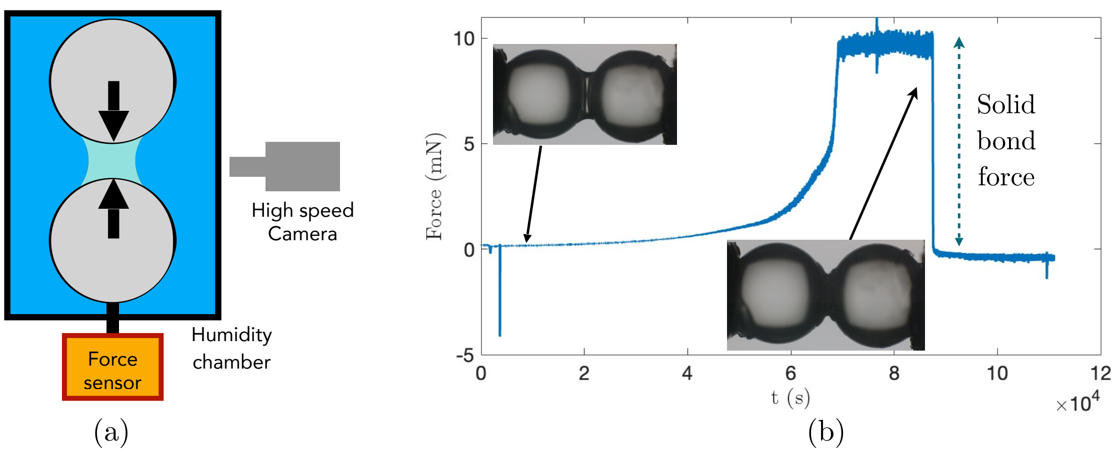


Figure 6: E(a) Schematic of the experimental setup developed in our laboratory to measure the capillary bridge force and the solid bond force after drying while imaging the capillary bridge. (b) Evolution of the force over time for a fixed distance between two glass beads and an initial capillary bridge of colloidal suspension leading to a solid bond.

We will measure the evolution of the inter-particle force over time and develop some scaling laws to evaluate the force as a function of the initial volume of liquid, composition, and size of the particles. Different formations of solid bonds will be considered: (i) drying of a capillary bridge in the presence of soluble species leading to a solid bond and (ii) capillary bridge between soluble particles leading to the sintering of the particles. The resulting inter-particle force will allow us to design more finely model aggregates that will be used in Task 1 and Task 2 to quantify the shear and energy leading to fragmentation. In addition, we will perform experiments where the particles come into contact and separate during the drying process, thus mimicking the process of drying and break-up.

We should emphasize that such spherical particles are, of course, not representative of industrially-relevant powders. We thus want to develop a *meso-scale* experimental approach where the two beads will be replaced by two inverted conical containers filled with initially wet powders [10]. The method will be to measure the force required to separate the two inverted conical containers filled with the powder under test. The bottom cone remains stationary, while the top cone is movable (similar to the experiments with beads). Initially, the open conical containers are brought into contact at their narrowest section of area A in a horizontal plane. The wet powder will then be poured from the top. Knowing the contact area and the force needed to separate the two surfaces will provide an estimate of the cohesive force per unit area of the wet powder, both during and after the drying process. Such an approach could then be compared to the results obtained with the shear-cell described in the following paragraph.

A "Rumpf's law" for solid bonds. The Rumpf's law describes the tensile strength of a granular material in terms of interparticle cohesion. It provides a macroscopic relationship based on the forces acting at the particle level. We want to study the evolution of this relation during the drying process.

We will thus extend our focus to a macroscopic granular system, initially wet (and up to the fully dry stage). We have recently acquired a shear cell (Anton Paar), which allows us to measure the macroscopic cohesive stress in wet granular materials and in granular materials where particles are connected by solid bonds (after drying). Leveraging these capabilities, we aim to consider a macroscopic relation (similar to Rumpf's law) at different stages of the drying process. The results gathered will be of interest for the different tasks of the project, but also at a broader scale, as highlighted in recent discussions with members of IFPRI. A key advantage is that measuring the macroscopic cohesion will be possible in fine grains and powders (such as calcium carbonate or powders used in the food industry).

Task 4. Drying under shear

The goal of this final task is to perform experiments similar to Tasks 1.1, 1.2, and 1.3 but now add the effect of drying to finalize the laboratory-scale tools. The work performed in the previous tasks will have provided a background of the actual shear imposed in the presence of grains, the ability to break up agglomerates without particles, and the resulting size distribution. We will then be able to add the effect of drying to finalize the project.

▷ **Task 4.1. Drying under shear of model agglomerates.** Using the three laboratory tools, we will investigate the drying of model agglomerates composed of spherical glass beads of small size, initially mixed with a liquid containing a dispersed solute. Upon drying, this solute will create solid bonds between the particles. Insights from Task 3 will guide us in determining the range of shear necessary to break these model agglomerates.

Thus, we will be able to study the effects of key parameters on the size distribution of agglomerates after drying to construct a regime map for the outcomes of drying wet powders under shear. The parameters include the imposed shear, the initial water content, the relative humidity and temperature, and the initial size distribution of grains.

Using the knowledge built in earlier tasks, we aim to develop models relating the cohesive energy E_c of the agglomerates to the breaking energy induced by shear. A particularly interesting point that we are looking forward to exploring is that the cohesive energy increases during drying, suggesting a potential interplay between the time scales of drying kinetics and the shear dynamics experienced by the system. Again, the different previous steps will allow us to compare those effects.

▷ **Task 4.2. Leveraging the tools: laboratory testing of industrial powders.** The final task of this project will involve applying the developed diagnostic tools to industrial powders, which will be provided by IFPRI members. We already have access to calcium carbonate powders and will seek additional samples.

For each powder, we will characterize the initial size distribution and conduct experiments by systematically varying the imposed shear, initial water content, and relative humidity and temperatures. We will then run experiments in the different setups to measure the final size distributions and mean sizes of agglomerates under different drying and shear conditions. We aim to develop simple models based on input parameters to predict the evolution of these characteristics and build a regime map.

> The main outcome of this task is to demonstrate the relevance of the diagnostic tools with model and industrial powders. The main result will be the size distribution and the mean size of the agglomerates under different drying dynamics and shear and simple models to account for their evolution with the input parameters.

5 CONCLUSION

A potential renewal of the support from IFPRI would allow us to develop this work and validate the relevance of these tools for both model and industrial powders. This work will provide industrial members with practical methods for optimizing the drying of their powders. In particular, they will have access to inexpensive laboratory-scale tools to perform quick tests before considering larger industrial-scale tests.

On a fundamental level, the knowledge gained will support further advancements in powder technology, benchmarking discrete element method (DEM) simulations, and improving our understanding of cohesive granular materials.

Tentative timeline. This proposal, resulting from different interactions with IFPRI members over the past two years, develops an ambitious and innovative plan to advance our understanding of cohesive agglomerates and the drying of powders under shear. While we aim to address all tasks, our primary focus will be to further characterize and develop the air-jet setup (Task 1.1), which enables high-shear conditions. This setup will be complemented by studies on the breakup of agglomerates by turbulent jets (Task 2.1), providing essential results on the threshold for fragmentation of the aggregates and on the resulting size distributions of the smaller agglomerates generated by the breakup. Task 3 will play a critical role in benchmarking our model cohesive agglomerates that will be used in Tasks 1 and 2. Finally, we will first focus on Task 4 on experiments using the air-jet setup to study drying scenarios under different conditions. In parallel, we plan to leverage the existing rotating drum setup, which is fully operational and capable of running long-duration experiments with minimal supervision. These experiments, which form our secondary priority, will provide additional insights into the dynamics of shear-induced drying.

Although of significant fundamental interest, the oscillating box setup represents a longer-term goal. Pending further discussion and a renewal of support, we hope to seek additional funding, possibly through NSF support (GOALI: Grant Opportunities for Academic Liaison with Industry) program.

The first two years of this project have been instrumental in discussions with various IFPRI members, helping us identify their specific needs related to drying under shear. During this period, we have designed and begun testing the experimental tools, laying the groundwork for the proposed research. We are confident that a renewal of IFPRI support for an additional three years will lead to substantial fundamental knowledge in powder technology and practical methods for industrial applications related to the drying of wet agglomerates under shear.

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