

1 Background

1.1 The importance of jamming volume fraction ϕ_J in solution processability.

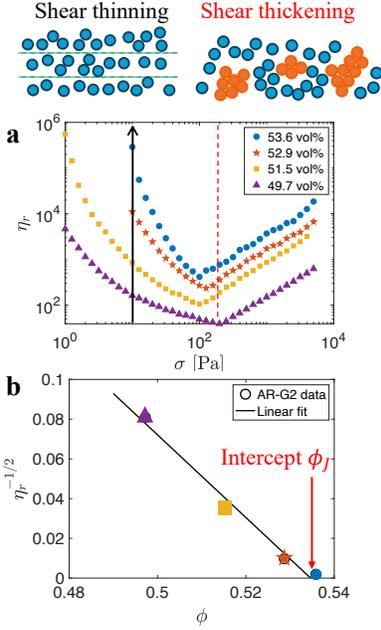


Figure 1. (a) Suspensions prepared with different solid content undergo shear thickening. (b) The conventional method of obtaining the jamming point is by extrapolating to infinite viscosity. Relative viscosity $\eta_r = \frac{\eta}{\eta_{\text{solvent}}}$.

suspensions. In practice, ϕ_J is commonly determined through a time-consuming process by measuring the shear viscosity of a suspension over a sufficient range of volume fraction ϕ_J . Each of these curves corresponds to a fixed volume fraction. A vertical cut through these curves reveals the point at which the viscosity begins to diverge (Fig. 1a). We may extrapolate for ϕ_J by fitting the resulting viscosity in different flow curves (Fig. 1b) to the Krieger-Dougherty equation [1]:

$$\eta = A \left(1 - \frac{\phi}{\phi_J}\right)^{1/2}.$$

During my postdoctoral training at the University of Delaware, I developed a strategy to rapidly probe the jamming volume fraction of an aqueous suspension (Fig. 2). The method was implemented using a stress-controlled rotational rheometer (MCR 500, Anton Paar, Graz, Austria) equipped with a commercial fixture consisting of a perforated bottom plate, from which a vacuum was applied. During the experiment, water exited through the bottom. The instantaneous concentration of the sample was determined by relating the height of the sample to its volume frac-

Multi-phase suspension is widely handled in manufacturing, and finding a low-energy route to processing is crucial to many industrial sectors. Rheology of a suspension is related to both the volume fraction [1] and the shear stress σ . Geometrical length-scales and mechanical properties in these systems diverge as the volume fraction ϕ approaches the jamming point, ϕ_J [2]. Close to the ϕ_J , a suspension can undergo shear thickening (Fig. 1a), which describes a drastic increase in viscosity [3, 4], as solid volume fraction approaches ϕ_J . For a system at rest, there is a unique transition to jamming. For instance, a system of monodispersed spheres reaches random closed packing at $\phi_J = 0.64$ [5,6]. In the flowing state, dilation, or an increase in volume of the suspension, may also occur as the particles move apart and push against the confining boundary [7]. The percolating structures can form at a lower volume fraction $\phi_J(\sigma)$. This is described by a model of particles existing in two states [8]: those experiencing high stress (f) are pushed together and cluster with a packing volume fraction ϕ_m that depends on the interparticle friction and applied stress σ [9], while those experiencing low stress ($1-f$) can rearrange into lanes, reaching a frictionless state with a higher packing fraction $\phi_0 = 0.71$ [10]. The resulting $\phi_J(\sigma) = f\phi_m(\sigma) + (1-f)\phi_0$ is a mixture of these two states.

In general, ϕ_J depends on surface asperities, roughness, and particle surface chemistry, and thus particle-level features can influence both microstructures under the flow and the rheology of

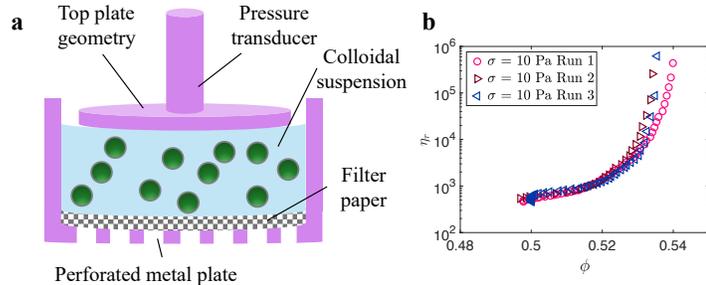


Figure 2. (a) Schematics for the immobilization cell geometry for probing particle jamming fraction under shear. (b) Dewatering paths during a typical experiment [4].

tion. As a proof of principle, I illustrated with an industrial titanian dioxide suspension, that a drastic reduction of both inter-particle friction and shear thickening could be achieved by grafting a lubricious polymer layer [4]. Nonetheless, the material requirements for this setup are rather substantial (10s of mL), making it unsuitable for designer materials, such as patchy particles [11], Janus dumbbells [12], and pharmaceuticals. Furthermore, this method does not provide direct visualization for structural insights, unlike techniques such as neutron scattering [13, 14].

1.2 High-throughput microrheology aids in formulation screening and design.

On the other hand, microscopy-based tools offer a promising way to visualize structure under the flow and provide a means to scale up. We have developed a microscopy-based, high-throughput microrheology platform integrated with machine learning-guided formulation design (Fig. 3).

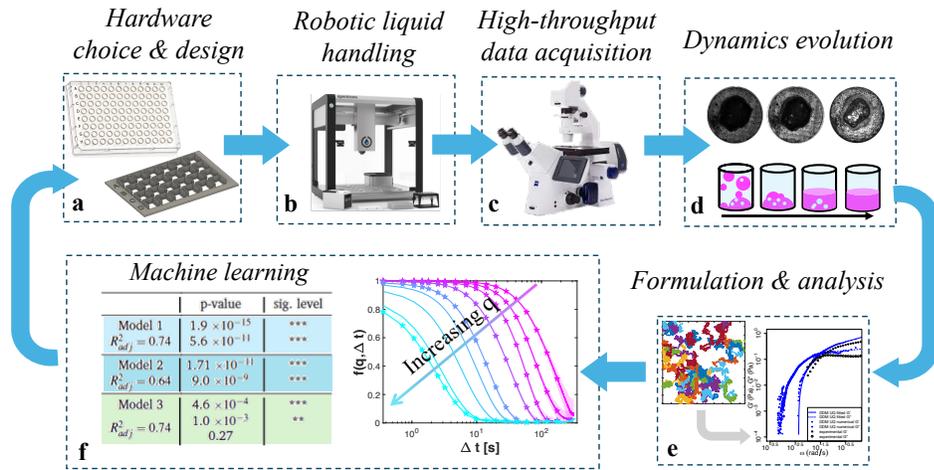


Figure 3. This workflow is realized by scanning through (a) 96-wellplates or 3D-printed custom geometries containing formulations mixed by (b) an automatic liquid handling system while (c) performing passive particle microrheology concurrently, to obtain the viscoelastic properties of evolving materials (d-e). (f) A mapping between phase behavior, formulation, and material properties can thus be established.

phase separation of two oppositely charged macromolecules [15]. This platform addresses two common obstacles typically encountered in processing complex fluids. First, there are a few ways to characterize material properties quantitatively as these processes occur. Second, the formulation space is often vast, while conventional characterization methods are low throughput and consume a lot of materials.

However, despite the success of microrheology in studying aqueous, low-viscosity fluids—making it ideal for examining high-value pharmaceuticals in the dilute limit—the applicability of this workflow to suspensions remains unclear. In particular, detecting small probe displacements in stiffer materials remains a challenge for passive microrheology, which relies on thermal fluctuations, so it cannot access certain material properties as compared to bulk rheometry. Developing high-throughput methods to investigate suspension properties will require reimagining existing practices. The key to eliminating the need for multiple loadings to measure high-shear viscosity at different volume fractions lies in extracting the solvent *in situ*, inspired by immobilization cell rheology. Furthermore, our experience with high-throughput microrheology provides a means to track the dynamic structural evolution and offers an analytical framework for understanding the structural changes associated with the jamming transition. Building on these past successes, we propose several experimental setups for high-throughput, *in situ*, rapid methods

This platform enables a high-throughput workflow to simultaneously characterize the morphology and mechanical properties of evolving multi-phase systems, in place of more cumbersome characterization methods. The experimental platform and computational workflow were applied to investigate a model system with a large parameter space: the rheology of polyelectrolyte complexes, from a liquid-liquid

for the measurement of the particle jamming fraction ϕ_J . The measured volume fraction will also be validated with *ex situ* methods. All these setups are designed with easy adoption for mass parallelization in mind.

2 Proposed research program

2.1 Model systems

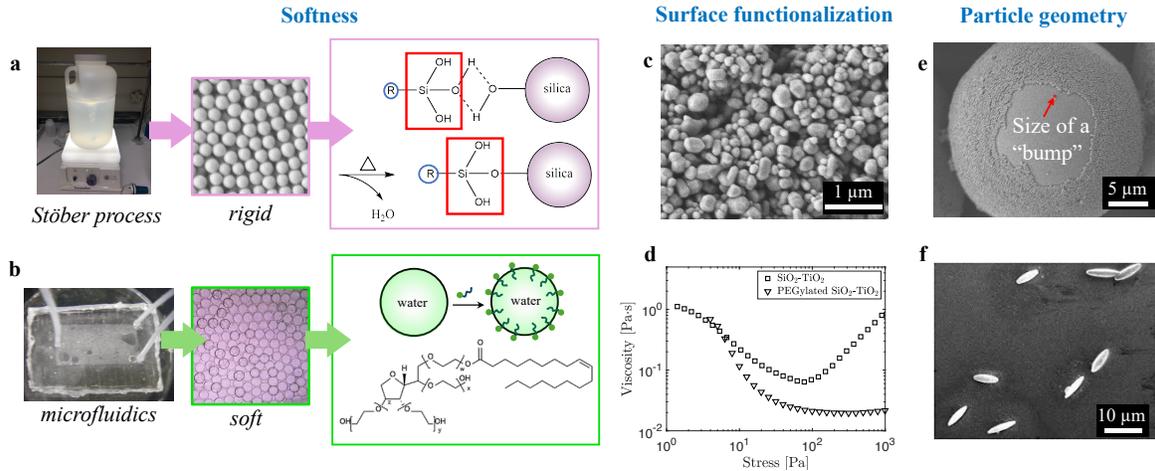


Figure 4. Methods to fabricate particles of different softness, surface functionalities, surface roughness, and shapes. Panel (a-e) are images from our own lab, and Panel (f) is derived from Ref. [16].

Many industrial formulations are composed of particles of heterogeneous attributes. For instance, household paint consists of pigment (hard) and resin (soft) particles, the manner through which these components segregate and dry ultimately determines the paint’s durability. Hence, experiments on ϕ_J must try to simulate these aspects as closely as possible. Particle systems with mixed rigidity, smooth or bumpy surfaces, defined surface functional groups, shapes, and varying degrees of polydispersity, can be created on demand. Our lab possesses a wealth of expertise in synthesizing both soft and hard particles as well as in surface treatment strategies in modifying their surface properties and roughness. These materials will be used as a starting point to validate the experimental setups we propose. Model hard particles will be silica particles made using the Stöber process (Fig. 4a), where particles ranging from 100s of nm to 1 μm are the most accessible. Model soft particles are made through the emulsification process, either by agitating (size range 1-20 μm) a water-in-oil mixture or by using microfluidics (Fig. 4b, size range 50-100 μm). This process results in either an emulsion (non-crosslinked) and hydrogels (crosslinked), with patch size and functionality that can be controlled by strategically decorating the interface with different surfactants. The competition between different surfactants and the temperature will result in distinct domain morphologies. Polyethylene glycol diacrylate can also be added to the water phase and subsequently crosslinked to make hydrogels of different softness (with moduli ranging from kPa to GPa). Surface patchiness will be introduced by a hydrolysis-condensation reaction using silane-terminated linkers (Fig. 4c). A lubricious polyethylene glycol layer is shown to significantly reduce shear thickening in titanium dioxide suspensions (Fig. 4d). By varying the annealing time and pH of reactions, the patch size and distribution can be controlled. By absorbing small colloids onto the surface of larger colloids, we may create particles with hierarchical surface features (Fig. 4e). Finally, by stretching commercial polystyrene particles in polymer matrices, we manufacture anisotropic, ellipsoidal particles (Fig. 4f). This research differs from prior efforts in that the impact of non-ideal particle-level features: heterogeneity and softness, will be investigated systematically

in detail. Hence, this work intends to fill fundamental and practical knowledge gaps that will delineate promising near-term particle design considerations.

2.2 Passive evaporation system

As discussed previously, ϕ_J depends on various particle-level features as well as the applied stress, σ . However, as a first approximation, determining ϕ_J under low-stress conditions provides an excellent starting point for extracting ϕ_J influenced by different particle-level characteristics. Toward this goal, we introduce our first setup, which consists of a central circular reservoir that is opened on both ends (Fig. 5), similar to the one showcased in [17]. These reservoirs are open on both ends, which enables solvent evaporation in controlled experiments. We will first establish a correspondence between *in situ* and *ex situ* volume fraction measurements using two independent methods. The *in situ* measurement is performed using optical techniques, which allow direct sampling and visualization of the number and size of spheres sedimented at the bottom of a suspension. The *ex situ* measurement relies on the capacitance method, which detects differences in permittivity between phases with varying solid fractions. Over time, solvent evaporation induces gradual compaction of the suspension volume and the decrease of interparticle distance. Our programmed, automated microscopy workflow will capture the structural evolution until the structure becomes arrested. The setup is scale-indifferent and expected to be applicable to both Brownian ($< 1 \mu\text{m}$) and non-Brownian (up to 10s of μm) particle sizes.

Image processing will be implemented by a Fourier-based image processing algorithm called differential dynamic microscopy (DDM), originally developed by [18]. Rather than tracking individual particle trajectories, this and related algorithms focus on understanding correlation decay through the intermediate scattering function. This approach is both less biased and more high-throughput, as it computes the transformation of image differences. The rationale is intuitive: if particle dynamics are fast, we expect the correlation between frames to decay quickly. In contrast, if the system becomes arrested and the structure remains frozen, the correlation between consecutive frames will persist for a longer time, with minimal decay. We have recently made several modifications to the original DDM algorithm [19,20] that greatly improve both speed and accuracy, in addition to providing an uncertainty quantification. Recently, the method has been successfully applied to understand the spatial modulations in the collective dynamics of a binary glassy system [21]. For simplicity, we will start with a pseudo-2D system and spread out the particles first in a monolayer, this would greatly reduce the complication and uncertainty associated with determining particle volume fraction. However, since our generalized image-processing framework does not rely on tracking particles, we envision that such a method could precisely pinpoint the moment of structural arrest in a 3D system just as easily. In that case, we could easily deduce the ϕ_J from the initial suspension concentration and the initial and final volume of the suspension. For verification, we will resort to using a confocal microscope to better capture the position of these particles.

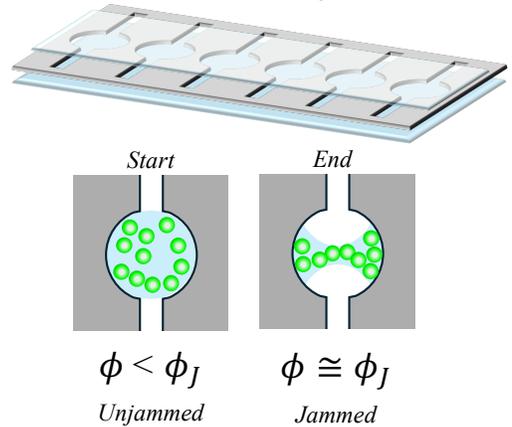


Figure 5. Top row: parallelize reservoirs allow sampling of multiple formulations simultaneously. Bottom row: a typical experiment starts with a suspension that becomes jammed with solvent evaporation.

System validation. We will start by verifying the random close pack limit of $\phi_J = \phi_{rcp} \approx 0.64$ for monodispersed hard spheres using this setup. Thereafter, we will systematically investigate sys-

tems with decreasing ϕ_J . One straightforward approach to achieve this is by increasing the surface roughness of the particles, which has been shown to enhance interparticle friction and promote shear thickening. Previous research, including our work [13] and simulations [9], demonstrates that increasing friction can systematically alter the macroscopic flow behaviors by promoting shear thickening. A straightforward path to create these particles has been reported in Refs. [22] and [23]. Following the protocol reported by [23], which relies on the electrostatic absorption of nanoparticles onto microscale silica particles, we have also successfully generated particles with controlled surface roughness in our lab (Fig. 4e). Additionally, verification of ϕ_J will be carried out by suspension rheometry, using the protocol specified in Fig. 1, and verification of initial volume fraction will be conducted using capacitance methods.

2.3 Flow in tapered channels

In the evaporation-driven scenario described in the last section, the flow is negligible, essentially absent, which prevents the exploration of flow-induced structure and flow-driven jamming.

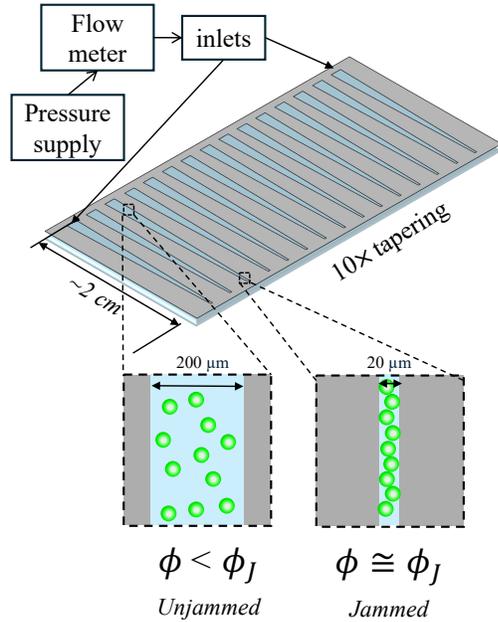


Figure 6. Top row: parallelize tapering channels driven by pressure at the inlet. Bottom row: blow-up view of the suspension state at the inlet and outlet.

To address this, we design a setup where flow in a slowly tapered channel can effectively concentrate particles. A similar setup was initially envisioned in Ref. [24], which focuses on clogging length. In this setup, the supplied pressure passes through a flow meter to ensure the same pressure at the inlet across all channels. In contrast to previous work, where the tapering angle is generally large the the total channel length tends to be small (100s of μm), in the reference work and our proposed work, the total length of the channel is several mm in total length. This longer channel length results in a much smaller tapering angle, such that each section of the channel can be approximated as having a constant width. The gradual tapering over the entire length of the channel effectively concentrates the particles by 10-fold. Furthermore, in contrast to Ref. [24], which focuses on clogging, we concentrate on jamming. To this end, we use submicron particles, at least 20x smaller than the narrowest sections of the channel, which are unlikely to clog or bridge the opening. Based on channel dimension, local flow rate, and initial particle concentration, we can easily compute the average shear rate and final ϕ_J where

jamming occurs. Furthermore, this setup places no constraints on the solvent type, thus it is applicable to both aqueous and nonaqueous suspensions, as the channels allow free flow of the exit stream. For this setup, it will be even more interesting to explore how the non-Newtonian nature of the solvent, such as a polymer solution, couples with the flow and influences ϕ_J .

System validation. While this setup can be used for single-component systems, it is especially useful for mixtures, particularly those involving both hard and soft spheres, where the soft particles deform in response to the driving flow. In past research, soft particles have been shown to be able to facilitate the flow of rigid particles in idealized geometries such as 2D hoppers [25]. Furthermore, since soft particles themselves can deform and pack at a higher volume fraction, we expect that with an increase in the percentage of stiff particles, jamming will occur at a lower volume fraction. A pseudo-2D system helps minimize heterogeneity caused by sedimentation during flow. Given PDMS boundary is mildly stretchable, particle migration and redistribution

are directly observable under dilational conditions. Additionally, multiplexing by repeating the same composition multiple times can further reduce variability, leading to improved precision in measuring ϕ_J . Our setup is expected to provide valuable insights into the complex interplay between deformation and component segregation under the flow that ultimately impacts ϕ_J and rheology.

2.4 Correlating force chain and the jamming

Connecting the microstructure of a suspension to its bulk rheological state is a long-standing challenge. Our third platform leverages digital image processing and machine learning to correlate the microstructure of the pre-jammed state with the resulting material properties. While lower ϕ_J has long been associated with force chains arising from interlinked colloidal structures due to interparticle friction [27], no systematic study has yet correlated the evolution of structure preceding the jammed state. This is partially attributed to the lack of direct imaging methods capturing the dynamics during shear. This can be achieved using stress-birefringent spheres, which light up when force is applied (Fig. 7). When viewed through crossed polarizers, these spheres reveal internal stresses by displaying distinct patterns of light. Due to our work with liquid crystals, our lab is well-equipped and has extensive experience in conducting crossed-polarizer microscopy. We will couple this capability with an optical shearing system (Linkam CSS450), which allows the structural dynamics of complex fluids to be directly observed under shearing conditions. We hypothesize that spheres immersed in the suspension will develop similar force chains under shear. This approach would allow us to map the development of the force chains for a suspension leading up to jamming.

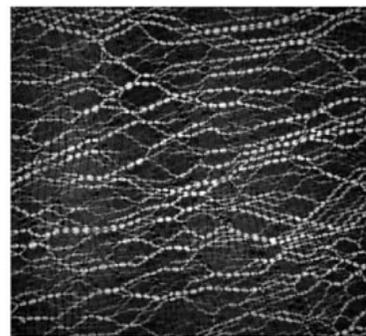


Figure 7. Observed stress pattern when a system of disks are placed under pure shear, adopted from Ref. [26]

3 Synergy with existing research programs

My group has several ongoing, complementary projects. The first is our efforts to understand microstructure using scattering, particularly for particles that are smaller than the optical wavelength and therefore difficult to visualize under the microscope. In our past work, small-angle X-ray scattering (SAXS) measurements have been used to characterize the anisotropic structure of hydrogel fibers, the anisotropic flow behavior of suspensions under shear, and the transition of Laponite clay suspensions from an initial fluid state to gelation. We have partnered with the BioPolymers, Automated Cellular Infrastructure, Flow, and Integrated Chemistry Materials Innovation Platform (BioPACIFIC MIP) at UC Santa Barbara, which is equipped with a state-of-the-art liquid gallium X-ray source. This high-brilliance instrument is essential for characterizing a large number of samples with precision. We have developed a shear cell, which can be used to capture the flow state of the suspension.

Another related effort investigates the microstructural evolution and rearrangement of hard tracers diffusing within a matrix of soft, deformable particles under quiescent and flow conditions, utilizing pseudo-2D, millifluidic devices. The primary focus is on understanding the non-Gaussian displacement of the tracers, which we attribute to the deformation or rearrangement of the matrix particles. To accomplish this, we employ model selection to determine the most appropriate model to describe the non-Gaussian dynamics. To uncover the underlying causes of these behaviors, we perform analysis on single-particle trajectories, create deformation maps, and compute velocity correlations. Ultimately, we will incorporate random and ordered pillar arrays into millifluidics

channels to explore the relationship between microstructural arrangement and suspension-level flow. These experiments are intended to fill fundamental and practical knowledge gaps and help delineate promising design considerations for soft and hard particle mixtures, including heterogeneous dynamics and clogging phenomena.

4 Support from IFPRI members

The primary benefit of this project lies in its ability to harness industry-leading capabilities in the surface treatment and modification of various particle systems through partnership with IFPRI members. Numerous industry partners have successfully produced engineered, surface-modified plastic, silica, and metal-based particles at production scales of several tons per hour, meeting the rigorous performance requirements of end-use applications. These standards and the resulting particles would be invaluable materials to incorporate into the research program, helping to elucidate the relationship between ϕ_J and the intended applications.

The student working on this project will also be exposed to industrial research and development with IFPRI members, which will enrich their education with exposure to a corporate R&D environment, its culture, and problems of interest. The student will be coached on important aspects of product development and decision-making involving a mix of assessments from practical implementation, and regulatory and market considerations, to more detailed strategies for meaningful product quality metrology and performance evaluations. Throughout this process, a clear understanding of the balance between pre-competitive research and proprietary efforts will be conveyed to ease complications in the public dissemination of results. Planned interactions with leaders, subject matter experts, manufacturing operators, and research personnel within the business at the corporate research facility and at local manufacturing plant sites will provide the student with insights into the structure and diversity within major particle technology firms.

5 Project Management, Timelines and Deliverables

This proposal will partially support one graduate student for three years. The graduate students will work on device prototyping, image acquisition, experiment design, and analysis. A tentative timeline and deliverables for each stage are presented to the right.

Objectives & Milestones	Yr25-26			Yr26-27			Yr27-28			Deliverables
	Fa	Sp	Su	Fa	Sp	Su	Fa	Sp	Su	
Aim 1: Construct model particle systems										A library of particles including size, shape and mechanical characterizations
Fabricate hard and soft particles	■									
Characterize industrial formulations	■	■								
Aim 2: Passive evaporative systems										A prototype capable of distinguishing between solids of high and low surface friction
Microfabricate channels		■	■							
Acquire images and system validation				■	■					
Image processing to obtain Φ_J						■	■			
Aim 3: Flow in tapered channels										A prototype capable of measuring jamming under different shear rates and dilational conditions
Microfabricate channels			■	■						
Set up and validate diff. flow rates				■	■					
Acquire and process images to get Φ_J							■	■		
Aim 4: Correlating force chain and the jamming										Software and platform to visualize and deduce network forces
Fabricate stress-birefringent sphere						■	■			
Force chain direction relative to Φ_J								■	■	

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