High Throughput Measurement of Particle Jamming: Response to reviewer comments

Daniel Hodgson | University of Edinburgh

Dear IFPRI Review Panel, thank you very much for the positive response to my proposal addressing the IFPRI Project Brief on High Throughput Measurement of Particle Jamming. Please find below my comments directly addressing the specific comments which were relayed back to me via Jim Michaels. Where appropriate I have also added/edited text in a revised version of the proposal to clarify the areas commented on.

1. "Most of the discussion focused on measurement and homogeneity of solids volume fraction. You don't actually specify how you'll measure the volume fraction, only that you'll identify jamming by a peak in the torque. Also, the device doesn't intrinsically promote homogeneity - especially on the "dry side" of the peak torque, where the device behaves like a granulator. (Hancock and York, whom you reference, used the device to study granulation "endpoint", which is a significantly heterogeneous system)."

The solid volume fraction will be calculated by converting the mass of the solids into a volume via the density, and then either accessing the volume of the liquid added directly when using e.g., a volumetric dispensing system, or by converting mass to volume via the liquid density, i.e., in the same way as volume fraction determination in samples prepared for rheological testing. The volume fraction is be defined as:

$$\phi = \frac{V_{\text{solid}}}{V_{\text{solid}}+V_{\text{liquid}}},$$

so above ϕ_{m} (the jamming volume fraction), by definition there will be inclusion of air, which is not accounted for in the determination of ϕ. Above this point (i.e., on the 'dry side' of the peak in torque or power), the system is heterogeneous on larger length scales than in a 'homogeneous' suspension (which is only homogeneous on length scales larger than the particle size, allowing us to define a bulk rheology). For $\phi > \phi_{\text{m}}$ we can still measure a 'bulk' material response, provided a representative portion of the sample is sheared between the mixer blade and wall (or other mixer blade depending on the equipment configuration). By definition, the size of the granules diverge at jamming and essentially become one large granule with the size of the system. The transition from dry powder (ϕ=1) to this point as liquid is added, essentially probes the combination of growing granule size relative to shear gap and the deformational properties of the granules as they are confined and sheared in this zone. As Hancock and York and others have found, the peak in mixer torque corresponds to the endpoint of granulation, which has previously been shown to correspond to ϕ_{m}.

As described at the start of Section 4, in all Work Packages, the mixer torque rheometer technique will be compared and benchmarked against the research laboratory standard rheometer-based measurements, which are outlined in Section 2 of the proposal. Whilst not high-throughput or widely available in all industrial settings, shear rheology still provides the best quantitative measurement of viscosity as a function of volume fraction (and thus identification of ϕ_{m}).

2. There was interest in whether you could make the measurement by adding powder to liquid vs. liquid to water. A corollary question is whether this would make any difference to the measured jamming fraction.

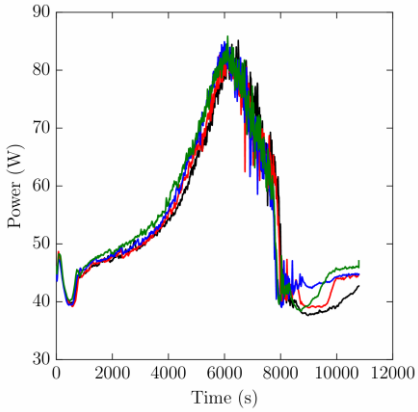
This point is specifically addressed in the final part of Work Package 2: "*In this WP we will explore any differences in mixer-torque rheometry from adding liquid to solid, or adding solid to liquid, thus starting to address some fundamental questions around the reconstitution of powders, and whether this experimental technique can add any new insight.*"

Whether it will make any difference to the measured jamming volume fraction is an interesting question and one which will be explored in the proposed work. In principle, for simple systems which have well defined primary particles which do not de-aggregate or break up under mixing stress, then ϕ_{m} should not depend on order of addition. However, as seen in Fig. 4 (c) and (d) of the proposal, more complex suspensions can have a rheology that depends on their shear history and preparation. For these systems, care would need to be taken to ensure that a stress similar to the process stress had been applied during sample preparation before making rheological measurements. One of the benefits of the mixer torque rheometer is that, by transitioning through ϕ_{m} from either above or below (i.e., adding liquid or solid, respectively), the

system must traverse the highest stress state possible (hence the peak in power or torque), and thus, the location should be agnostic to either addition method. Ambiguity or error could arise if only a subset of measurements were taken from either direction and ϕ_m inferred from fitting some function to these data (in exactly the same way for the interpretation of the data in Fig 4 (c) of the proposal).

3. Finally, how does baseline noise affect the measurement (if at all)?

Yes, in principle, baseline noise could affect the measurement, although in practice this depends very much on the mixer being used and the specific sample being measured. For most industrially relevant suspensions (i.e., dense suspensions with very high viscosity), the baseline noise in power draw is usually insignificant compared to the additional power required to mix typical dense pastes. The data below shows a repeated measurement of power as a function of time for a planetary mixer combining an industrially relevant food system comprising non-spherical particles and very high size polydispersity. The data show that for four repeat measurements, whilst there is some noise in the data, the overall analysis (here, main peak position and height) is the same each time.



One area where careful attention to the noise will be taken is in determining whether the power draw is steady as a function of time (which in turn indicates whether the microstructure of the powder-liquid mixture is stable and has reached a steady state). For each new system, this will need to be assessed once, to understand how complex the system is and how much time is required. For simple, non-aggregated systems, the system will reach steady state once the added liquid or solid has been evenly distributed throughout the system, however for more complex systems, reaching steady state may take longer if all of it needs to pass through the high shear zone in order to, say, break up aggregates into primary particles. In an industrial setting, where the type of particle and other ingredients are constant for a given formulation, then this process will only need to be done a very small number of times. Following an initial calibration with a new material, a protocol could be defined and the measurement of ϕ_m can be made without further system testing.

The role of suspension/granule microstructure in generating this noise will be explored beyond year 3 (as detailed in the Outlook section of the proposal) since there has been some work previously done linking the periodicity of these fluctuations to powder saturation and granule structure (Hancock et al., 1994). However, this understanding does not directly influence the principal aim of this work which is developing a high-throughput methodology for determining ϕ_m.

I hope that these comments address your questions and concerns adequately. If there are any further clarifications that need to be made in order to fully understand the programme of work, or other ways in which the proposal can generally be improved, please do let me know.

Best regards,

Daniel Hodgson
Director of the Edinburgh Complex Fluids Partnership
University of Edinburgh, UK