High Throughput Measurement of Particle Jamming

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1. Why is ϕ_m an important quantity in industrial processing of dense suspensions?

In dense particulate systems, the jamming volume fraction, ϕ_m, sets the upper bound on solids loading below which homogeneous flow is possible. At higher solids loading, the system state transitions to a jammed solid which may granulate or fracture in response to mechanical stress (Fig. 1a) [1]. For many systems, including extrusion of ceramic pastes and pumping of chocolate or concrete, ϕ_m represents the maximum solids loading achievable before processing failure or material defects occur. The rheology of dense suspensions is also controlled by 'distance to jamming', i.e., $\Delta\phi = \phi_m - \phi$, with the viscosity and yield stresses increasing dramatically as $\phi \to \phi_m$. Control over these rheological parameters is essential for many applications including the mouthfeel of liquid chocolate or the processability of concrete. The relative viscosity, η_r, of a suspension, i.e., its measured viscosity normalised by the binder viscosity, η_s, is well described by a Krieger-Dougherty type equation [2] for a very wide range of particulate suspensions (e.g., Figure 1b), and in which ϕ and ϕ_m are key control parameters:

$$\eta_r = \frac{\eta}{\eta_s} = \left(1 - \frac{\phi}{\phi_m}\right)^{-\lambda}. \quad (1)$$

Being able to optimise formulations to either give consistent rheology (i.e., consistent $\Delta\phi$) or being able to maximise solid loading so that e.g., the amount of water used in a ceramic paste can be minimised, thus reducing processing energy, is important, and requires a careful quantification of ϕ_m.

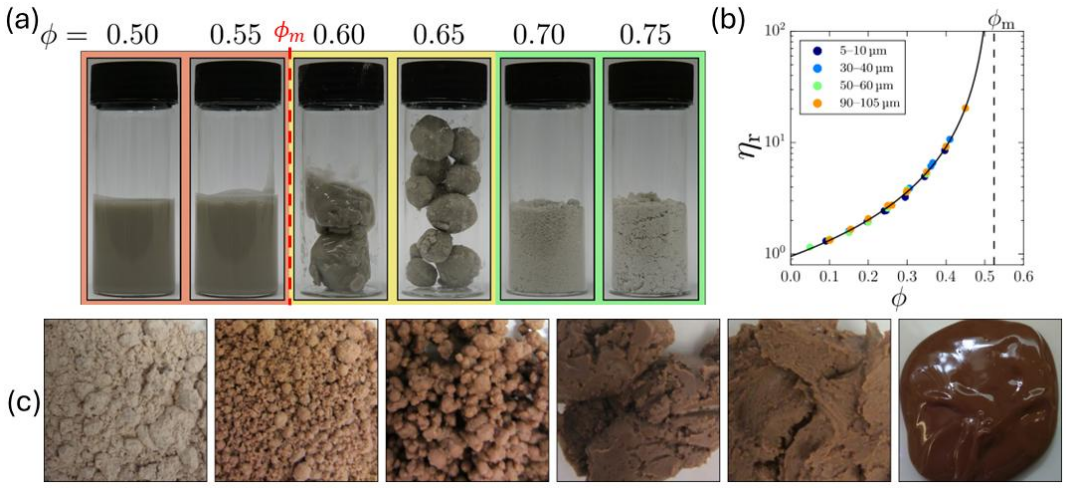

Figure 1: (a) Final mixture state for a model system of glass spheres in water, transitioning from flowing to jammed solid at $\phi > 0.55$ (figure adapted from [1]). (b) Relative viscosity as a function of volume fraction fitted with the Krieger-Dougherty equation (data replotted from [3]). (c) Transition from granules to fluid during chocolate conching (figure adapted from [4]).

In other industrial applications which focus on drier, granulated systems, ϕ_m also represents the point at which the system becomes 'over wet', no longer forming stable granules, but instead transitioning to a slurry or flowing suspension thus determining the scope of formulation space and in some cases influencing the properties of the granules formed [5]. In other industrial processes, this drier state is necessarily traversed as either liquid content is increased during reconstitution of powders, or as ϕ_m is modified in e.g., the conching of chocolate (Fig.1c) [4].

In all such applications and processes, ϕ_m is critical to determining the end-state rheology, the process energy used, and therefore the success or failure of manufacture. The jamming point varies with particle shape, size polydispersity, inter-particle interactions, often mediated by process additives, and so batch-to-batch variability is a common challenge when formulating close to ϕ_m. Therefore, a robust, high throughput methodology to measure ϕ_m is needed by industry to characterise incoming materials and rapidly reformulate.

In Sec. 2 we outline current methods for determining ϕ_m and their limitations, then in Sec. 3 we outline how mixer-torque rheometry offers the clear potential for an accurate high-throughput measurement of ϕ_m. This technique is well suited to more complex industrial systems, the multiple stress-scales of which we illustrate in Sec. 4 and in which we detail how complexity will be progressively increased and explored in Work Packages 1 to 5. Finally, in Sec. 5 we detail how this project leverages existing expertise and capability within the Edinburgh Complex Fluids Partnership and broader soft matter group.

2. Current methods for measuring ϕ_m and their limitations

There are many ways to quantify ϕ_m or to detect changes in the jamming point. Being able to accurately measure and quantify ϕ_m is essential during reformulation if trying to maintain constant rheological properties. For established formulations running in production, it may simply be enough to detect changes in ϕ_m, requiring a technique sensitive and precise enough to detect these changes.

In this section, the existing methodologies for quantifying ϕ_m or inferring changes indirectly are reviewed, and a comparison table summarising these techniques is provided at the end.

Shear rheometry: This is the canonical, laboratory-standard method for obtaining an estimate of ϕ_m. The process involves preparing suspension samples at various different ϕ and measuring the rheology. Often the high shear viscosity, η_∞, is normalised by the solvent viscosity, η_s, to obtain the relative viscosity, η_r, which is then plotted as a function of volume fraction. These data are then fitted using the Krieger-Docherty equation (Eq. (1)) to obtain a value for ϕ_m [6]. Whilst this technique produces accurate determination of ϕ_m for a broad range of systems, it is time consuming and requires expertise to load complex samples and gather reliable rheological measurements.

Atterberg plastic & liquid limits: These tests (ASTM D4318) were originally developed for testing fine-grained soils and involve rolling or tapping powder-liquid mixtures, until some pre-defined shape fails. The plastic limit defines where a sample no longer has sufficient moisture to be thinned down to a cylinder of 3.2mm, whereas the liquid limit defines a point at which a split sample will fluidise and flow together under some standardised deformation. Both tests are empirical, and neither precisely locate ϕ_m, although they bound its value.

There are a number of other techniques which are useful for determining ϕ_m in simple model systems, but are challenged by sample complexity. **Sedimentation/centrifugation** consists of preparing a suspension at volume fraction, ϕ. The suspension is then left to sediment under gravity, or centrifuged at high speeds to generate higher particle pressures. The final sediment height is then measured and the volume fraction can be directly calculated [7]. While this method is undoubtedly high throughput and centrifuges are accessible at relatively low cost, for systems of attractive particles or systems with a yield-stress binder, the gravitational stress must overcome the yield stress of the system; for some systems this will require extremely large centrifugal speeds which will not be achievable in commercial equipment. **Immobilisation cell rheometry** uses a shear cell in which the lower plate is permeable to the liquid binder phase but constrains the solid particle phase [8]. The liquid is removed from the shear cell whilst sheared, thus increasing the volume fraction. The system is sheared at a constant shear rate as ϕ increases, until the viscosity diverges. A similar set up is the **capillary-stress controlled rheometer** or the "Capillarytron" [9]. Both systems could be used as a high throughput measurement of ϕ_m for larger particles suspended in a very inviscid fluid, but for small particles, systems with high polydispersity, or high viscosity or non-Newtonian binders, the pressure required to remove the fluid phase is extremely high. Luo et al. removed fluid from the system via evaporation, which for water or other volatile solvents is straightforward, but challenging for oils or polymeric solutions.

There are also a number of techniques which could be useful for detecting changes in the jamming point, even if they are unable to directly quantify ϕ_m without significant calibration. These include **tap density** which can give an estimate of the maximum packing of the dry powder, which in an ideal system is closely related to ϕ_m. However, this technique is not suitable for complex systems with high polydispersity or aspect ratio, or systems that are agglomerated and require high shear to break them up. Measurement of **single particle friction coefficient (μ)** can also be used to detect potential changes in particle-particle friction coefficient, which in turn can affect ϕ_m [10]. This method, however, is not high throughput, and cannot easily be utilised for polydisperse or irregularly shaped particles which are common in industrial processing. Another way of investigating changes in interparticle friction coefficient is via a submerged **angle of repose** experiment. This technique is capable handling polydisperse and irregular particles, but analysis is challenging for attractive suspensions, and likely not possible for complex non-Newtonian binders.

Discrete element modelling & simulation: Another method for estimating ϕ_m is using modelling and simulation, in particular Discrete Element Modelling (DEM) [11]. Suspensions at varying ϕ can be prepared and sheared to obtain η_r. These data can be fitted using Equation 1 and an estimate of ϕ_m obtained [12]. Whilst relatively quick to perform and a wide range of variables easily explored, it is challenging to accurately determine ϕ_m due to accurately characterising powder properties (size distribution, shape, interparticle interaction) as inputs to the simulations. Very high polydispersity can also pose a computational scale issue since the number of particles needed for a representative distribution grows very rapidly as the distribution widens. For every single $10\mu m$ particle, a thousand $1\mu m$ particles are needed for an equivalent volume.

Technique	High throughput	Widely available	Accurate	Precise	Complex systems
Shear rheometry	×	×	✓	✓	✓
Atterberg plastic & liquid limits	✓	✓	×	×	✓
Sedimentation/centrifugation	✓	✓	✓	✓	×
Immobilisation cell rheometry	✓	×	✓	✓	×
Capillary-stress controlled rheometry	✓	×	✓	✓	×
Tap density	✓	✓	✓	×	×
Single particle μ	×	×	✓	×	×
Angle of repose	✓	✓	✓	×	×
Simulation	✓	✓	✓	×	×

Table 1: Summary of the different techniques used to quantify ϕ_m or infer changes indirectly. The only technique which offers accurate quantification in a range of complex systems is shear rheometry, which is not high throughput and requires expertise in making and interpreting measurements. Other techniques may be high throughput and widely accessible to industry, but are not suitable for use on industrial complex systems.

3. Investigating mixer-torque rheometry as a high throughput method for determining ϕ_m

In this project we will investigate mixer-torque rheometry as a method for determining ϕ_m in a range of industrially-relevant systems of increasing complexity. A mixer-torque rheometer is essentially a mixing device which measures the torque during the mixing process. In its simplest manifestation such a device simply measures the power draw during mixing, providing the mixing rate is kept constant and the power-torque relationship is monotonic and ideally approximately linear (not a gearbox-equipped motor). Such devices are widely available, spanning low-cost kitchen mixers coupled with consumer-grade power meters, through to laboratory research equipment, including twin screw extruders or Brabender mixers.

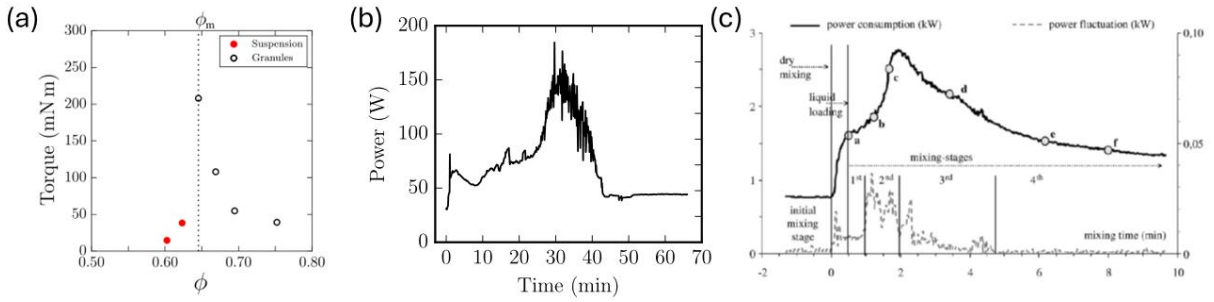

Figure 2: (a) Mixer torque vs volume fraction. Figure interpreted and replotted from [13], as per [5]. (b) Power draw from a planetary mixer during chocolate conching. Figure replotted from [14]. (c) Cement mixer power as a function of time shows a similar peaked curve as for chocolate indicating similar underpinning physical mechanisms [4].

Mixer-torque rheometers have indirectly been used to explore the transition from suspensions to granules (i.e., locating ϕ_m) previously by Hancock et al. in their work on wet granulation [13]. Hancock et al. used mixer-torque rheometry to explore different substrate-binder interactions for a model system of glass ballotini (1-20μm) in low viscosity polymer solutions (PVP and HPMC) at a range of different liquid contents (i.e., ϕ). In the paper Hancock et al. plot the mixer-torque vs liquid content (reproduced in figure 2a here as a function of ϕ). They interpret this peak torque curve as corresponding to different granulation states, from 'powder' at very low liquid content through 'pendular' and 'funicular' to 'capillary' state once the powder is fully saturated. Any additional liquid causes the system to transition to an 'over wet' droplet state in which the system is no longer granulated but is instead a suspension [15]. In effect the transition from capillary state to over wet droplet state identifies ϕ_m. In terms of the torque response, the capillary state corresponds to the peak, with further addition of liquid resulting in a rapid reduction in measured torque.

Mixer-torque, or more precisely mixer-power, has previously been used to assess the granulation to suspension transition in complex industrial systems. In researching the conching of chocolate, Blanco et al. used a kitchen planetary mixer with attached power meter to study the conching of chocolate from a dry powder state through to flowing suspension (Figure

2b). In other work a similar setup was used by to study the concrete mixing process by Cazacliu and Roquet [14]. They observed a similar peak power curve and again identified this peak as a transition from solid to liquid state (Figure 2c).

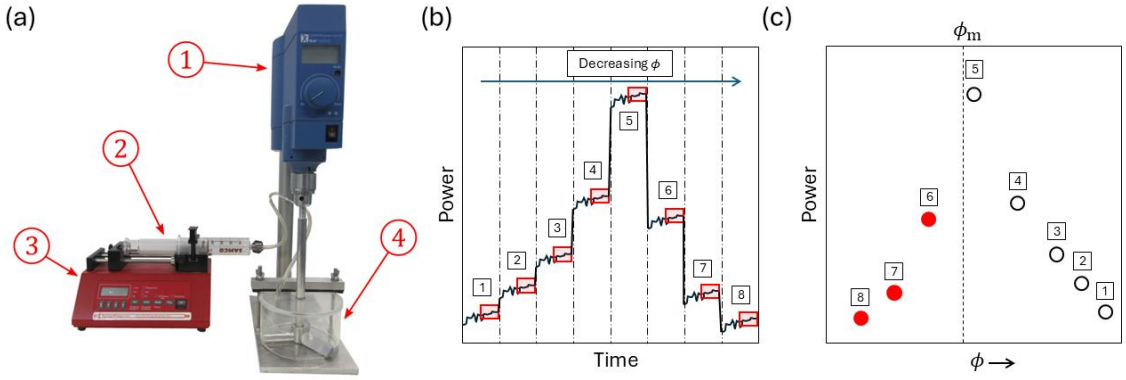

Figure 3: (a) Schematic of a previously used mixer for batch granulation [1]. The set-up mixes dry powder whilst liquid is added using the syringe pump system. A power meter attached to the overhead mixer could report as a proxy for how viscous the contained mixture is at a given volume fraction, ϕ. (b) Expected form of power vs time raw data from the mixer torque set up. Dashed lines indicate liquid addition points, thereby decreasing ϕ. Red boxes indicate a period of averaging, which then form the data in part (c) which plots power vs ϕ, thus enabling ϕ_m to be identified by the position of the peak.

By staged addition of liquid, Fig 3a (above), mixing torque at controlled decreasing ϕ, Fig. 3b gives ϕ_m from peak power, Fig. 3c. Volume fraction is determined by converting powder and liquid mass to volume via their material densities. This combines the precision and accuracy of multi-sample rheology with the high throughput nature of plastic limit testing, whilst avoiding some of the limitations of the other techniques outlines in Sec. 2 when applied to complex industrial systems.

4. Proposed research overview & work packages

The overarching aim of this project is to develop an industry-adoptable high throughput methodology for measuring ϕ_m and detecting changes in ϕ_m due to varying feedstock, formulation, or processing conditions. The first part of this project will be to demonstrate proof of concept on simple model systems to validate that the methodology can be used to accurately measure ϕ_m with benchmarking against known methodologies for determining ϕ_m, principally shear rheometry, as described in Sec. 2. Of course, this technique needs to be used across a range of much more complex industrial formulations, taking into account particles of different sizes, high polydispersity in size, multi component systems, or binders with complex rheology. Therefore, subsequent work packages focus on more complex systems.

System complexity & stress scales

For simple systems of granular (diameter $\geq 10\mu\mathrm{m}$) hard particles in a Newtonian binder, the suspension rheology of a sample at $\phi < \phi_\mathrm{m}$ is Newtonian, i.e., constant viscosity as a function of applied shear stress or shear rate (Fig. 4a, ●), and this viscosity follows the Krieger-Dougherty behaviour, Eq. (1), increasing rapidly as the system volume fraction approaches ϕ_m. However most industrial systems are not so simple, often exhibiting complex rheological behaviour such as shear thinning or thickening, being composed of complex particle shapes, sizes, and chemistries, or being suspended in binders that are inherently non-Newtonian. For such systems there are additional stress scales, and the applied stress, σ, relative to these determines the rheology and thus mixer-torque data. For example, in shear thickening systems (Fig 4a, ●) below a critical onset stress, $\sigma^\star$, particles can slide past each other and thus the viscosity is lower. For $\sigma > \sigma^\star$ particle contacts become frictional, the distance to jamming $\Delta\phi$ is reduced and the viscosity increases. For shear thinning systems (Fig 4a, ●), attractive particles can form a percolating microstructure with a yield stress, σ_y; when $\sigma < \sigma_\mathrm{y}$, the system does not flow.

Another critical stress scale is the fracture stress, σ_f, which scales as Σ/d, where Σ is the fluid surface tension and d the particle diameter [16]. When $\sigma \geq \sigma_\mathrm{f}$ the particles start to protrude through the liquid surface (Fig. 4b) and as the stress increases, the free surface can break up, limiting the stress scales accessible in conventional rheometry. Further stress scales of relevance to industrial systems and processing are those of aggregate break up, σ_agg, or primary particle breakage, particularly relevant for friable minerals. Some systems can additionally have a background binder yield stress, $\sigma_\mathrm{y}^\mathrm{b}$, which, like the particulate yield stress, must be exceeded by the applied stress before a system can flow.

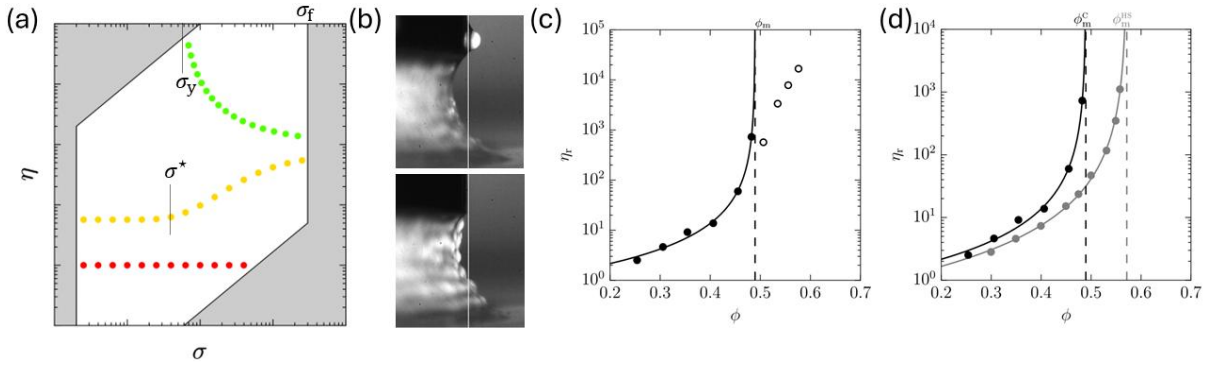

Figure 4: (a) Representative viscosity (η) vs shear stress (σ) for a Newtonian fluid (●), a shear thickening fluid (●), and a shear thinning fluid (●). The greyed out region is inaccessible in standard rheometry due to torque limitations of the rheometer, the fracture stress (σ_f) of the sample, secondary flows or inertial breakup of inviscid samples, or limits on the length of the experiment time at low shear rates. (b) Upper panel shows a dense suspension at rest, and lower, same under shear, now exhibiting dilatancy, i.e., $\sigma \gtrsim \sigma_f$ [16]. (c) A dense suspension prepared via centrifugation. At $\phi > \phi_m$, the system should be jammed, however, here the system can flow due to aggregate break-up, thus increasing ϕ_m. (d) Comparison of the same sample, one prepared by centrifugation (C), one prepared using a high-shear mixer (HS). The two preparation methods diverge with different ϕ_m indicating a different aggregate state.

The magnitude of these stresses relative to each other and to the applied stress are critical to characterising ϕ_{m}. For example, for a simple system of glass spheres in glycerol/water prepared by centrifuging the particles into the liquid phase to remove the air and then homogenising using a gentle roller, one can use multi-sample rheometry to quantify ϕ_{m} (Fig 4b ●), however at higher volume fraction ($\phi > \phi_{\mathrm{m}}$), rather than granulate, suspensions with finite viscosity can be obtained (Fig. 4b ○). Underpinning this behaviour is a breakdown of glass particle aggregates formed during the centrifugation preparation process when the handling stresses at high-ϕ exceed σ_{agg}. One can apply a high stress ($\sigma \gg \sigma_{\mathrm{agg}}$) prior to rheometry and obtain a higher value of ϕ_{m}, corresponding to the two particle aggregate states (Fig. 4c). In manufacture, where processing stresses, σ_{proc}, can be high, one must ensure that the mixer-torque rheometer set-up is achieving the same stresses, relative to the other key stress scales in the system, as described above.

A detailed breakdown of the first 3 years of work across 5 different Work Packages (WPs) is detailed below. The length and timing of these WPs are detailed in Table 2 below.

Work Package 1: Mixer Torque Rheometry for Industrial Systems

WP1 will focus on basic equipment setup using a range of available mixers including kitchen planetary remixes and overhead mixers (e.g., Ika Eurostar Power Control-Visc) coupled with power meters. A simple model system will be used to confirm the findings of Hancock et al., with conventional shear rheometry of the same systems run in parallel to confirm ϕ_{m} and provide a quantitative comparison. Experimental protocols to add liquid using a syringe pump, thereby decreasing ϕ, during mixing will be developed, with consideration given to ensure added liquid is distributed homogeneously throughout the powder. The system complexity will be extended beyond simple spherical particles using quartz flour in a similar size range. This has similar chemistry to the glass particles but is expected to pack differently and thus give different ϕ_{m}. The difference between a simple model system and a system with shape complexity will be quantitatively compared.

Work Package 2: Increasing Particle Complexity

In WP2 we will use the equipment and protocols developed in WP1 to explore systems with higher complexity and more industrial relevance. In this WP we will prepare samples with different particle diameters, d, spanning several hundred nanometres through to hundreds of micrometres. Having characterised a number of different sizes spanning this range, we will then sequentially mix these together to produce particle size distributions with high polydispersity. We can also modify the model system to explore the role of particle-particle attraction/adhesion either by silanising this silica/glass surfaces or by switching the aqueous binder for an oil-based alternative. 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.

Work Package 3: Investigating Binder Complexity

In WP3 we explore the role of binder complexity replacing the simple Newtonian fluids used in WP1 and WP2 for those which exhibit shear thinning or yield stress behaviour, which could be made-up of a background dense suspension of attractive particles with significantly smaller diameter, or a polymer solution with increasing concentration. The purpose here is to increase the yield stress or viscosity of the background fluid and assess the utility of mixer-torque rheometry in determining

ϕ_m for systems where a significant proportion of the applied stress may be borne by the suspending matrix rather than the particle network.

Work Package 4: Assessing Dynamic Systems

WP4 tackles the most complex industrial systems; those which can dynamically change during the mixing process. Changes can take place due to a range of different mechanisms (e.g., compositional changes due to evaporation of solvent, or chemical changes activated by the mixing process energy) but here we restrict ourselves to structural changes in primary particles or particle aggregates, which can lead to changes in ϕ_m. Examples of such changes include attrition and breakage of soft particles in e.g., talcs, the break-up of high aspect ratio particles, or de-agglomeration, for example in the conching of chocolate [4] (see Fig. 1c or Fig. 4b).

Work Package 5: Validation via Industrial Round Robin

WP5 is a 'round robin' activity which will run in parallel to WPs 2-4, once the mixer-torque capability is established and has been proven for a simple, well-controlled model system. In this WP, IFPRI members will be invited to send samples for testing on the mixer-torque set-up, with validation of ϕ_m, where possible, using shear rheometry. The industrial samples will ideally also build up in complexity in line with the developments in WPs 1-4. The main purposes of this WP are to *(a)* gain a better understand of industry requirements around measuring ϕ_m, and *(b)* accelerate learnings from this programme into IFPRI membership, beyond the fixed engagement points at the AGMs and reports. Where companies are not able to send samples to Edinburgh either for cost, confidentiality, or material safety reasons, we will still engage directly, and where possible assist companies to set up their own testing apparatus in-house. Data confidentiality around this WP will be discussed on an individual company or sample-by-sample basis to maximise collective learning whilst maintaining strict confidentiality around proprietary information.

	2025	2026				2027				2028		
	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3
WP1												
WP2												
WP3												
WP4												
WP5												
				AGM	**Report 1**			AGM	**Report 2**		AGM	**Final Report**

Table 2: Overview of how the 5 WPs fit together over the first 3-year period, with the annual report schedule.

The overarching deliverable of this 3-year programme of work is a robust, accessible methodology with which IFPRI members can quantify ϕ_m for a range of complex industrial systems. The methodology will have been tested across a range of model and member-supplied systems, and will have been validated against standard laboratory best-in-class methodologies. Progress will be disseminated to wider membership in-person at the IFPRI AGMs and through annual written reports. Through the work in WP5, proactive engagement with a number of interested member companies will ideally see this methodology adopted and tested in a range of industrial settings. We also anticipate publishing the findings of this work in relevant, high impact journals, e.g., Powder Technology.

Outlook beyond 3 years

Beyond this 3-year programme of work, the next steps are to further explore linking quantitative data from mixer-torque rheometry to the bulk state and microstructure of dense suspensions ($\phi < \phi_m$), and granules or pastes with a significant air void fraction ($\phi > \phi_m$). Metrics such as the periodicity of power fluctuations and the amplitude of these oscillations have previously been linked to saturation and granule structure [13]. Understanding the link between power consumption and dynamically changing ϕ_m (e.g., conching) also direct potential processing efficiencies and energy savings, or optimisation of other reconstitution applications. Through consultation and feedback from the IFPRI membership, coupled with learnings from the first 3-year programme, the scope and deliverables of a potential renewal will be finalised in the final year of an initial term.

5. Synergy with existing expertise & research in Edinburgh

The Edinburgh Complex Fluids Partnership (ECFP) is the knowledge exchange organisation for complex fluids research at the University of Edinburgh. We are based in the Soft Matter and Biological Physics group of the School of Physics and Astronomy, and partner with the Schools of Chemistry, Engineering and Biology to deliver our mission of being a leading centre for science-driven formulation across multiple sectors, through consultancy services, collaborative research, and partnership development. Since its inception in 2012, ECFP has worked with more than 55 companies, ranging from small local manufacturers to global blue-chip companies. Dr Daniel Hodgson is the Director of ECFP and has a scientific background in dense suspension rheology and the transition to granulation, with a particular focus on industrially relevant systems, having completed postdoctoral research projects in collaboration with Mars Chocolate and Corning Incorporated before his current role leading ECFP.

More broadly, the soft matter physics group in Edinburgh has a long history of working with dense suspensions and colloidal dispersions, and has deep expertise in understanding how particle-particle interactions and micro-physics give rise to a rich phenomenology of rheological behaviours, with active researchers including Professor Wilson Poon, Dr John Royer, Dr James Richards, and Dr Rory O'Neill. The group has a range of research activities related to dense suspensions and granular materials, ranging from PhD projects studying how robots move in grain silos to personal care formulations and battery slurries, through to Masters, Senior Honours and Industry summer students studying industrially relevant systems including food products, concrete pastes, and suspensions for additive manufacture.

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