



IFPRI

International Fine Particle Research Institute

Powder & Liquid Reconstitution Workshop

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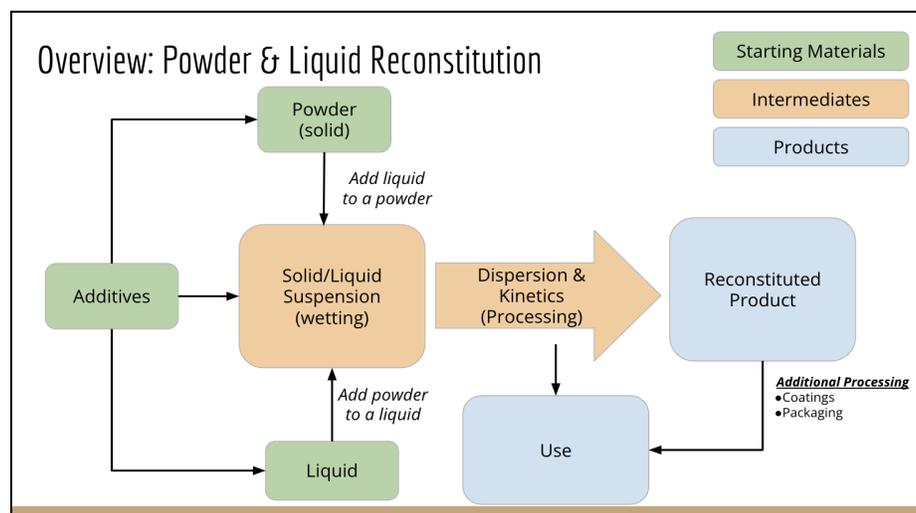


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Introduction

Problem Statement

In industry, a disconnect often exists between powder suppliers (manufacturers) and end users (customers) when dealing with reconstitution. Manufacturers may modify production processes to optimize yield, enhance safety, reduce costs, or scale up production. While such changes may produce powders that meet specifications, such as particle size distribution or specific surface area, they can inadvertently alter the powders’ behavior when combined with liquids – a process referred to as **reconstitution** in this report. These subtle changes in physical or chemical properties can lead to challenges for end users, who rely on stable reconstitution outcomes. Such challenges impact the performance of products across industries and affect markets worth billions of dollars annually. For example;

1. **Facial cleansing products:** These include creams, pastes, and scrubs that often require mixing powders with liquids during manufacturing. This market, recently valued at \$13 billion is projected to grow to \$21 Billion by 2032, driven by the demand for natural, organic, and non-toxic “clean beauty” products [1]. Top global manufacturers of these products include Estée Lauder, Procter & Gamble, Johnson & Johnson, L’Oréal, Unilever, and others.
2. **Lithium Ion Batteries:** Battery manufacturing involves slurry preparation by combining powders and liquids, followed by coating the slurry on foil to make an electrode. In 2023, the lithium ion battery market was valued at \$54 billion, and is expected to reach \$182 billion by 2030, fueled by the growth of electric vehicles, grid energy storage, and consumer electronics [2]. Top global manufacturers include CATL, LG Energy Solutions, Panasonic, SK Innovation Co, Samsung SDI Co, Tesla, and others.
3. **Pasta Manufacturing:** Producing pasta involves mixing flour with water to create dough, which is then processed into noodles. This market, estimated at \$68 billion in 2023, is forecasted to grow to \$100 billion by 2032, driven by demand for gluten-free, vegetarian, and ready-to-eat products [3]. Barilla, Campbell Soup Company, Unilever, the Kraft Heinz Company, and Nestle are notable manufacturers of these products.

These examples represent only a fraction of the industries reliant on powder-liquid reconstitution. Other sectors include pharmaceuticals, catalysis, petrochemicals, agriculture, polymers, construction materials, food and beverages, and more. The total

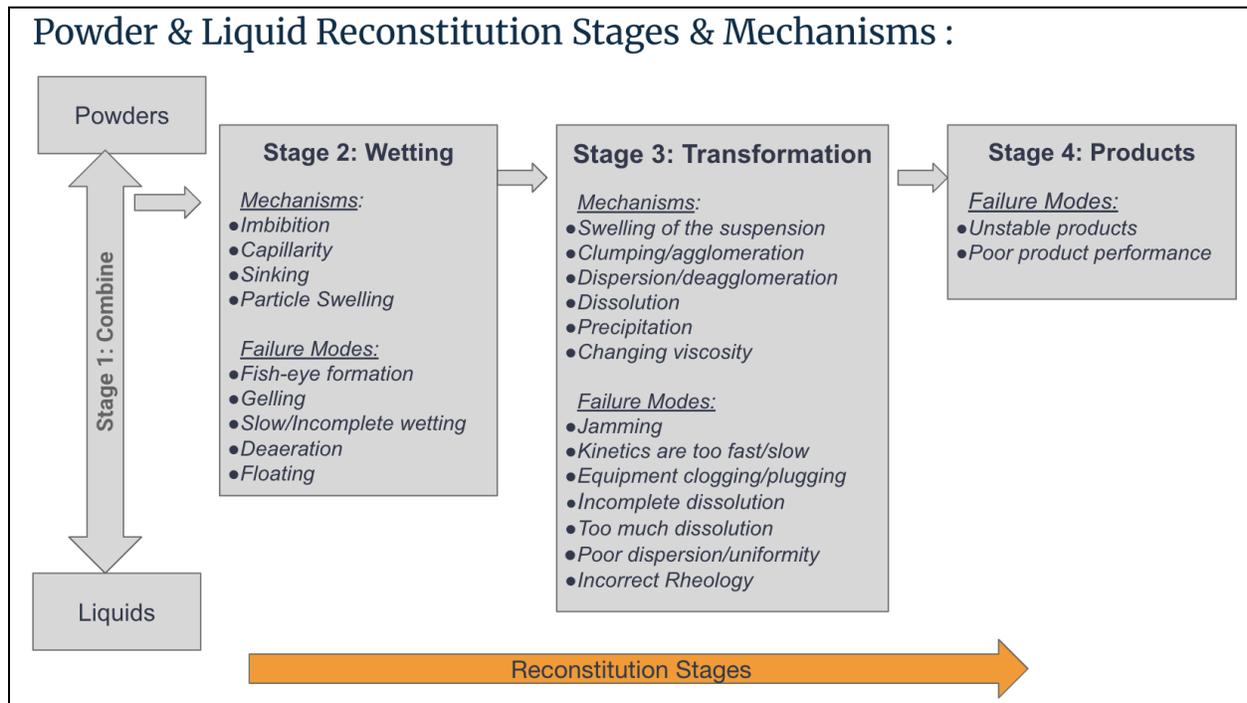
addressable market for reconstituted products likely exceeds hundreds of billions of dollars, underscoring the critical need for further research into optimizing powder-liquid processes to capitalize on these opportunities.

Reconstitution Definition

The workshop planning committee's definition of reconstitution covers a wide range of powder materials and the processes by which they generate an acceptable intermediate or final product when mixed with liquids.

Reconstitution Stages and Mechanisms.

To obtain a reconstituted product, powders must be mixed with liquids to form a suspension or solution. During this process, multiple stages and mechanisms occur as the starting materials are combined and transformed into the final product.



The first stage of reconstitution is to **combine liquids and powders**. This can be done by adding liquids to powders or, conversely, powders to liquids. The order of operations and the equipment used significantly impact the outcome of the reconstitution process.

The second stage is **wetting**, where liquid is incorporated into the solid, displacing the gas present in the system. Imbibition is a mechanism that occurs during wetting, in which the liquid penetrates between powder particles, displacing gas volume. Capillary imbibition, or capillarity¹, refers to the displacement of gas within a particle by a liquid,

¹ More broadly, capillarity refers to phenomena where three or more phases interact at their respective interfaces. The corresponding interfacial surface energies give rise to contact angles where three of them meet, and to pressure differences across interfaces that separate two of them, typically predicted by the Young-Laplace equation. In the

affecting the intraparticle pore volume. As a result, particles often experience changes in volume or density, leading to swelling or sinking. Once these mechanisms have completed, the powder-liquid mixture has fully undergone the wetting stage.

The third stage of reconstitution is **transformation**, where energy is added to the system. Examples of energy application include mixing, shearing, adding or removing heat, adjusting pressure, transferring materials, and packaging. The magnitude of energy applied, and the equipment used can significantly impact the performance of the reconstituted product.

Once transformation is complete, the resulting suspension or solution is the final **product**—the fourth stage of reconstitution. This stage does not involve distinct mechanisms or failure modes. Instead, failures typically observed in earlier stages manifest when qualifying or testing the transformed product. These failures often result in product instability or failure to meet performance standards.

It is important to recognize that reconstitution processes are highly complex, and the stages and mechanisms vary with each specific case. In many instances, the stages occur simultaneously. Given the dynamic nature of these systems, applying a first-principles approach to model and predict reconstitution outcomes is challenging. To drive innovation in reconstitution processes, it is essential to fund research that addresses the gaps and failure modes at each stage, ultimately resulting in more reliable models and better products.

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Workshop Objective

The objective of the workshop was to bring together a broad range of expertise, spanning academia and industry, to discuss challenges and gaps industrial IFPRI members experience in powder and liquid reconstitution. During the workshop, we planned to:

1. provide context and examples of the challenges experienced in industrial reconstitution processes;
2. discuss advances in the state-of-the-art research in materials science, processing, and associated powder & liquid reconstitution characterization;
3. identify gaps and resources needed to overcome the challenges in scaling powder/liquid reconstitution processes.

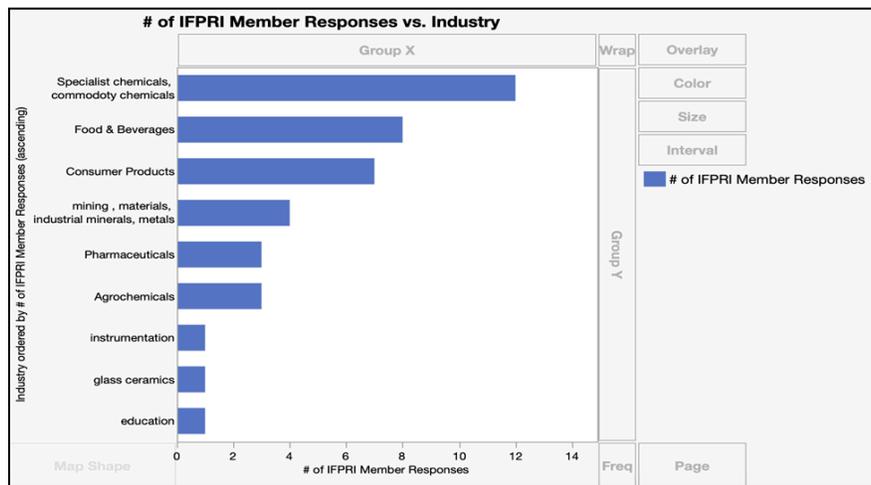
context of reconstitution, this can apply when solids enclose both gas and liquid. But it could also arise when two liquids are present around solids, for example oil fat, water and particles.

Through the workshop, we intended to define scientific questions that IFPRI and other agencies should promote to fund research projects in the field. This report summarizes our findings.

IFPRI Member Survey of Reconstitution & Results

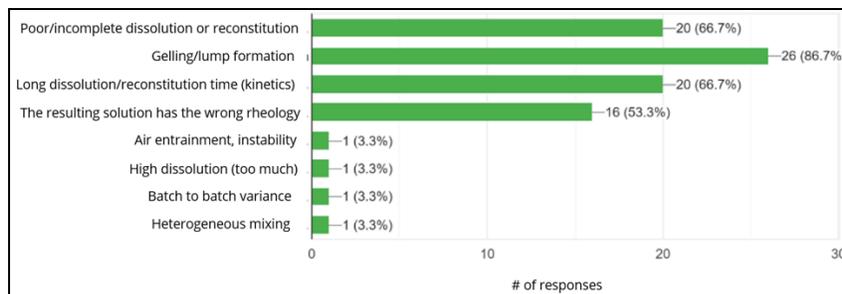
To gauge interest and the industrial importance of reconstitution processes, a survey was sent to IFPRI members. The questions were aimed to develop, understand, and quantify the gaps and needs of reconstitution in relation to the processes, technical knowledge and characterization tools.

A total of 30 unique responses were recorded from a variety of industries: with 50% of the makeup from the specialist chemicals and food and beverages sector.

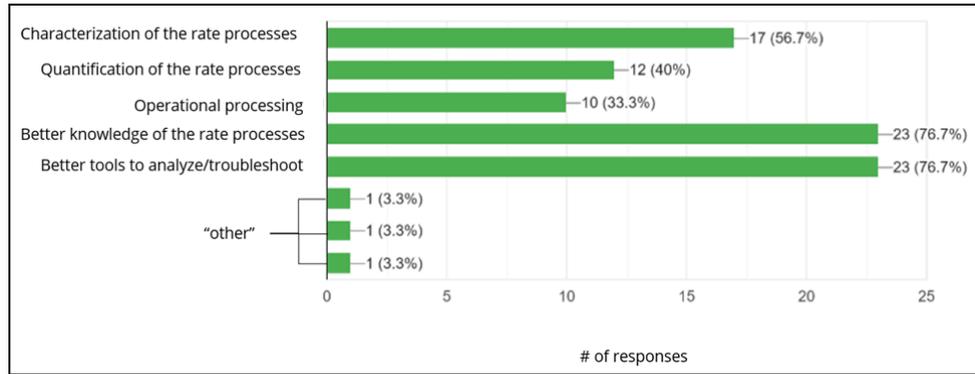


Reconstitution is clearly an important process for nearly all respective businesses: over 90% of respondents indicated that reconstitution is key for their activities. Of these members, over 80% indicated that they were either not sure or suggested that poor technical understanding was a leading cause for poor reconstitution outcomes.

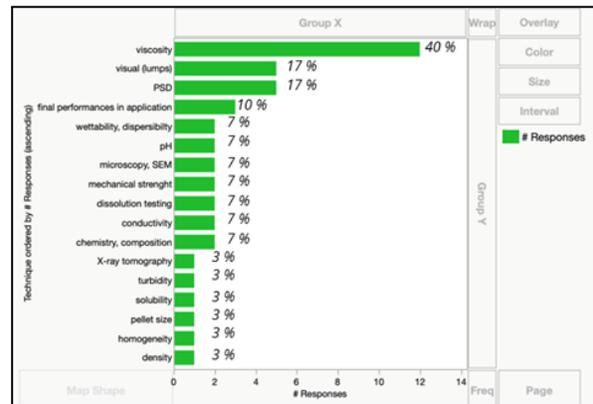
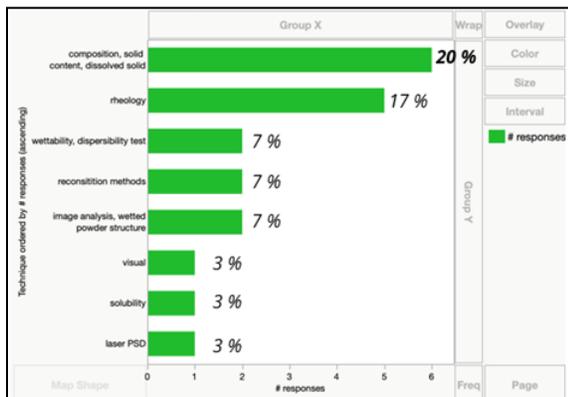
The typical reconstitution issues that members often encountered were: poor total dissolution, slow reconstitution times and the formation of viscous gels and fish eyes. Other issues found are also shown on the figure below.



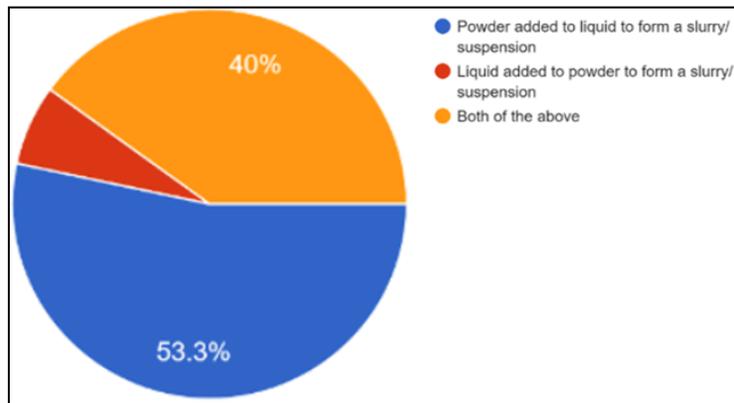
Such issues were challenging to predict primarily due to a lack of understanding of main mechanisms. Additionally, they are described to be hard to characterize; where many members attempt different characterization approaches with most not providing significant support or outcomes to root-cause issues. The following figure describes areas where IFPRI members require more research and development.



A question was asked on the current scope of tools and techniques available to the current IFPRI members. The answers showed that most of the techniques currently used focus on evaluating the final reconstituted products; specifically the viscosity, rheological properties and visual lump formation.

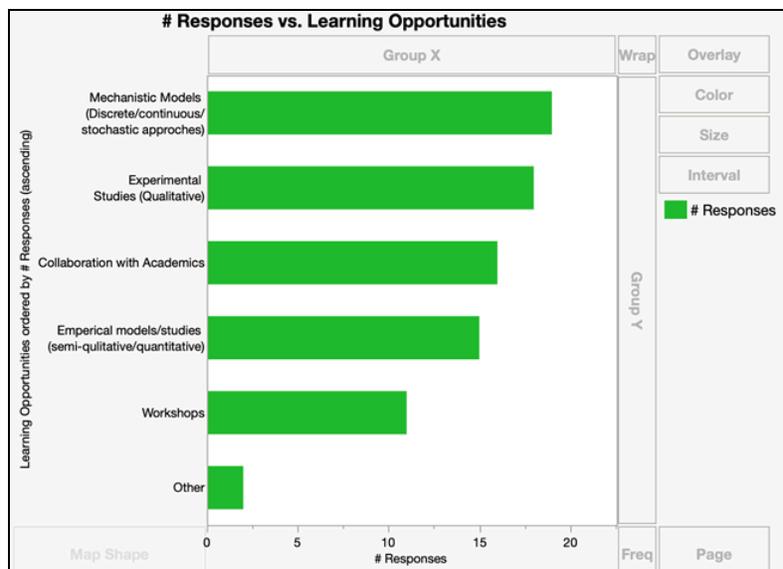


Most members check rheology and composition during processing because the methods for characterizing these properties are easy to execute on-site. A wide range of measurement tools and techniques can be used, but IFPRI members have not aligned on a “gold standard” approach to quantify the critical properties to evaluate success in reconstituted products and processes.



A majority of IFPRI members deal with both liquid added to powder and the opposite, but it is clear that powder added to liquid is the most common form. This suggests that characterizing processing, especially *in-situ*, can vary significantly depending on the powder-liquid addition approaches.

Finally, we asked IFPRI members what research IFPRI should fund. As the figure below illustrates, most members would find mechanistic models, supported with some experimental work, to be most valuable. There is a strong contingent of members who believe that empirical models would be valuable, which highlights the current state of knowledge within the field of reconstitution.



Key takeaways of the survey

1. IFPRI members struggle with the behaviors of solid/liquid transformations and understanding the underlying mechanisms. Industry is not able to effectively predict and mitigate undesirable attributes that arise during the transformation process.

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2. There is a limited knowledge of techniques to characterize the transformation process. As a consequence, process scale up is difficult, long, and requires significant cost/time to complete.
 3. There is broad acceptance that IFPRI members are not fully aware of best methods to characterize the rate processes related to reconstitution.
 4. Current literature does not address the appropriate studies or quantitative validation that can help support issues faced by IFPRI members.
 5. There is a strong belief among IFPRI members that, when appropriately guided, academic research can significantly improve the issues faced within reconstitution processes in industry.

Workshop Session 1: Industrial Applications

The objective of this session was to set the scene from an industrial perspective. Some concrete industrial issues and challenges encountered by food, chemical, agricultural or material science companies in the area of powder reconstitution were shared with the academics to provide a basis for discussion in the breakout session.

Technical Presentations:

Crop Protection Water Dispersible Granules – Formulation Development and Launch Troubleshooting.

Brian Levy-Polis, FMC.

Water dispersible granules are a crop protection formulation involving multiple wetting steps from start-up to point of use. Final product specifications related to the user experience along with specialized test methods will be discussed. Correlations where particle size distribution, paste water content, and mixture melting point will be shown how many variables through the entire production process can affect final performance.

Brian Levy-Polis discussed powder wetting in the manufacture and use of granules for crop protection. Nearly all types of crop protection formulations involve the wetting of an active ingredient. Water Dispersible Granule formulations start as powders, are wetted into a paste, extruded and dried, and finally dispersed in water by the consumer. Quantitative tests exist for wettability and determining the quality of granule breakup, but scaling up from development to pilot to plant is often based on observed correlations and visual cues that are subject to significant variability, requiring on-the-fly or post-launch adjustments when launching new products.

Science of Reconstitution: Recent Progresses and Upcoming Challenges.

Laurent Forny, Nestlé.

Dehydrated beverages continue to present significant business opportunities and show continuous growth [1]. As a result, the science of reconstitution has garnered substantial interest from both the academic and industrial sectors over the past two decades, as

evidenced by the steady rise in scientific publications and patents [2,3]. This collaborative effort has led to the partial filling of scientific gaps identified in 2009 [4]. Notably, significant progress has been made in understanding wetting, capillarity, and the dispersion of soluble powders. In this presentation, we will provide a summary of our most recent achievements in the field [4-6]. However, challenges persist, particularly in light of new product innovations driven by major consumer trends such as organic or sugar reduction. These innovations often require an expanded scientific understanding and customized reconstitution performance. We will discuss several examples that highlight upcoming challenges, including the reconstitution of functional ingredients (e.g., tastants, encapsulated bioactives) in complex products (e.g., fat-based matrices) and within complex environments (e.g., the mouth, digestive tract). An objective for the industry is to establish a comprehensive link between physical properties and reconstitution performance. By addressing these challenges and advancing our scientific knowledge, we could further enhance the development of dehydrated beverages and meet the evolving demands of consumers.

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Challenges in Reconstitution of Infant Milk: What Do Customers Do, And How to Measure It?

Martijn van der Hoeven, Danone.

For premium infant milk formula (IMF) products, consumers (in China) can pay up to EUR 45 to EUR 50 per can of product. For this amount of money, they expect perfectly behaving products that dissolve instantly. However, for manufacturers it is difficult to know and predict the way consumers use their products. A key example is the type of bottle used for preparation, there are many varieties available with different dimensions which could influence the reconstitution process.

Also, consumers prepare bottles in different ways. Some people shake very hard to (hopefully) accelerate the reconstitution process and have a bottle ready quickly. Others

shake or swirl very gently to prevent foam formation. The temperature of the water is also another very important parameter, influencing kinetics of the powder dissolution. There are cultural differences influencing this parameter, with water temperatures varying depending on the country of use. On social media, many different pieces of advice are shared, which can change frequently. Overall, IMF manufacturers must ensure proper dissolution under a series of different circumstances, to ensure limited lumps and foam formation.

In the manufacturing process and products design stages, the powder is fine-tuned at four different levels:

1. Bulk powder properties determine the bulk volume (and thus the weight of product per scoop), the homogeneity and the reconstitution.
2. The bulk powder is made up of agglomerated particles blended with additional ingredients. The size of the particle and the degree of agglomeration determine porosity, which in turn influence bulk powder properties.
3. The primary particles determine the agglomerate structure and the moisture content. Where a moisture content that is too low inhibits proper reconstitution.
4. Within a primary particle, the location of fat droplets, as well as the degree of free fat determines the hydrophobicity of the powder.

Manufacturers must design and produce the powder with all these aspects in mind to produce a perfect powder. This must be done in machines which are the size of large buildings and where the fresh powder does not behave in the same way as aged powder.

Fundamental understanding of the mechanisms that drive good reconstitution, including all the different sub-processes involved, is therefore key. Only once we understand this properly, we can then design and manufacture particles that behave as intended, even if a consumer deviates from the preparation instructions on the product label.

The Reconstitution Enigma of Nonmetallic Slurries.

Marty Murtagh, Corning.

Abstract: The reconstitution enigma of nonmetallic slurries emerges because of a lack of developed methods for reconstituting non-aqueous Stereolithography (SLA) additive manufacturing (a.k.a. Vat photopolymerization) slurry product waste when compared to an aqueous diluent precursor product. SLS uses a UV light source to cure a slurry into hardened plastic or inorganic composite. Reconstitution of the non-aqueous photopolymerization slurries compromises final product green strength (post heat treatment) due to the inability to reform a consistent polymerized 3D printed network.

Marty Murtagh discussed the constitution of a SLS slurry feed stock consisting of a reactive diluent (aqueous or non-aqueous), non-metallic particles, photopolymerizable

monomer, photo-initiator, and dispersant. The initial slurry rheology has a preferred viscosity of $<20\text{Pa}\cdot\text{s}$ at a shear rate range of 10 to 100s^{-1} requiring a balance between particle size and solid loading for a stable slurry, the viscosity and UV depth of penetration. The slurry viscosity increases exponentially with the increase in solid content - at equivalent solid content, the smaller the particle size, the greater the viscosity. UV depth of penetration and critical exposure decrease with increasing solid content due to greater attenuation. It is for these reasons, the non-aqueous SLS reconstitution enigma exists.

Manufacturing Dense Inorganic Solids Suspensions: Industrial Challenge Case Study.

Mingzhe Yu, Johnson Matthey

High solids content poses significant challenges in the manufacturing processes involving inorganic fine powders and liquid batching. As the solids concentration increases, powders tend to form layers and experience caking, making it difficult for the materials to integrate smoothly when transitioning from low to high solid content. Additionally, fine powders are prone to agglomeration, leading to a reduction in bulk density and further complicating dispersion within the liquid medium.

Mingzhe Yu presented an industrial case study, the suspension of inorganic solids at high concentrations was examined, highlighting how fine powders can float and form agglomerates as solids content rises. Various impeller designs were tested to evaluate their effectiveness in incorporating powders into the liquid phase. For solids contents below 40%, no substantial difference was noted across impeller types, with the exception of the sawtooth impeller. However, an optimized pitch blade turbine impeller demonstrated superior performance, achieving solid contents up to 50% with significantly reduced incorporation time. Notably, the study found no transitional regime between turbulent and laminar flow, with high power numbers persisting in the laminar regime.

Breakout Session and Reporting Back

The objective was to discuss the main challenges and gaps faced by industries when dealing with powder reconstitution, *i.e.* transforming granular materials into their intended functional forms (liquid or paste) by adding liquid to them or adding them to liquid. The main gaps and challenges can be categorized in 5 groups:

1. System & Process Complexity and Multidisciplinary nature of phenomena at play :
 - The reconstitution process is complex, involving multiple phases (air/water/solid, mechanisms, scales, and dynamics).

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- It requires expertise in various disciplines, including soft matter physics, formulation science, interface science, chemistry, and processing, to bridge the gap between product design and manufacturing effectively.
 - Collaborations between product designers and formulators and process engineers should be considered in early stage of product development to benefit from respective knowledge and expertise

2. Technological, Methodological & Modeling limitations and issues:

- There are significant gaps in characterizing the starting (dry powder) materials (e.g., pore structure, particle surface interactions), the reconstitution process itself and its dynamics (measuring interactions between water and particles or predicting dissolution rates).
- There are also gaps in characterisation of solvent, as besides rheology, measurements on liquids are often limited.
- Gaps in characterisation are often compounded by lack of mechanistic knowledge of how these quantities individually or in combination affect the reconstitution process and its outcome.
- Existing academic studies often use simplified models that are not directly inspired by industrial practice or limited techniques, making them less relevant for applications.
- It is challenging to accurately measure and analyze data from reconstitution processes, where simplifying complex data into single parameters may result in information loss.
- Developing relevant models that can capture the complexity of reconstitution, including the physics and dynamics of the process, is an ongoing challenge.
- The complexity of these processes, coupled with the limited capabilities of computational models, makes it difficult to have access to deep understanding.
- The limits of current knowledge and/or methodologies make it sometimes difficult to identify / measure all the factors influencing reconstitution processes (what is not known cannot be measured).

3. Understanding of the phenomena at play:

- Specific physical phenomena such as particle wettability, clump formation, capillary bridges, and the role of particle properties (e.g., roughness, size polydispersity, microporosity) are not well understood.
- This lack of understanding hampers the ability to predict and optimize processes like mixing, dispersion, and dissolution.

4. Diversity in Applications and Use Cases:

- Different applications, from dilute temporary suspensions to concentrated pastes, products for consumers or for manufacturing purposes, require a much deeper understanding of the kinetics and quality of the final product.
- Challenges in scaling up processes, predicting energy and time requirements, and addressing the unique needs of different product types remain significant.

5. Standardization and Consistency:

- Ensuring consistent product performance, especially for consumer applications where reconstitution cannot be tightly controlled, is a significant challenge.
- Developing standardized manufacturing processes to ensure products' quality across different environments, including various solvents (aqueous vs. organic) is therefore needed but requires to know the key parameters driving performances & final functionalities.

Overall, the industry faces substantial challenges in standardizing, characterizing, and understanding the complex and multidisciplinary nature of powder reconstitution processes. These challenges span from fundamental scientific understanding to practical application in manufacturing and consumer use.

Workshop Session 2: Fundamentals of wetting powders

We define powder wetting as the displacement of air by liquid on a powder internal & external surface. It is a critical and necessary step in the journey of reconstituting a powder into a liquid (or vice versa) and it is influenced by properties of the solid, like size, shape, roughness, degree of agglomeration and hydrophobicity as well as by the characteristics of the wetting liquid.

The objective of this session was to look in detail at the different mechanisms & forces involved in powder wetting, serving as the fundamentals upon which improvements in terms of the rate of powder wetting can be explored.

Technical presentations:

Capillary Imbibition: Progress in Understanding Powder Wetting Phenomena and Applications.

Mathis Fricke (TU Darmstadt). Joint work with Joël De Coninck, Université libre de Bruxelles.

Capillary imbibition plays a key role in many industrially relevant processes, ranging from microfluidics to oil recovery. Because fully resolved CFD simulations of the process are typically very costly, there is a strong demand for simplified models (typically

ordinary differential equations - ODEs) that can be solved efficiently and enable advanced methods such as optimization and control of the flow in complex situations. A famous example of such a simplified model is the Lucas-Washburn equation (1), which dates back to the 1920s. Also, the wetting of powder beds is commonly described with such an approach. In particular, the Lucas-Washburn equation with an effective contact angle based on the well-known Cassie-Baxter equation is used to predict the imbibition into the heterogeneous powder beds (see, (2)).

In this talk, we outline a modeling framework which allows deriving an ODE model of the capillary imbibition by modeling the various dissipative mechanisms in the system (3). The resulting models are (by construction) consistent with the second law of thermodynamics and are shown to agree very well with experimental data by Quéré (4). As a model system for the wetting of a powder bed, we revisit the classical Lucas-Washburn model for a chemically heterogeneous surface composed of two materials with different equilibrium contact angles. We show that the imbibition time depends on the distribution of the two materials within the tube. Remarkably, we can show that the Cassie-Baxter equation does not provide the correct averaging of the wettability to match the imbibition time. Instead, another type of averaging is necessary. This result may have significant implications for maximizing the imbibition speed into a heterogeneous material such as a powder bed (publication in preparation).

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Grains at Interfaces - Principles and Applications to Liquid Marbles.

David Quere (ESPCI Paris).

Surface energy generally makes grains stick to interfaces, which was exploited to stabilize emulsions, as first proposed by Ramsden and Pickering. Grains can also decorate water/air interfaces, resulting in original properties. Liquid marbles, namely drops surrounded by solid particles (in the range of 10 to 50 μm), hardly interact with their substrates, in sharp contrast with usual drops. They adhere very little (with a solid-like friction) and they have remarkable mobility when subject to a force, for instance gravity, due to the reduction of their contact with the substrate. Their properties are even more spectacular when the liquid is viscous (glycerol), in which case their speed is enhanced by a further (dynamic) reduction of their contact. All these properties make liquid marbles especially suited to transport efficiently small amounts of liquids, especially when they are viscous.

Sparse Fiber Structures to Minimize Powder Mass and Maximize Rehydration and Mixing.

Pat Spicer (University of New South Wales).

There is a trend in consumer product development to increasingly concentrate or "compact" products to remove water and adjust product form accordingly. This saves transport costs and increases sustainability but presents significant issues with hydration and stability of the reconstituted products. Many of these issues stem from the formation of complex microstructures, with difficult to mix rheology, as a result of surfactant and polymeric structural and phase transitions. Study of these kinetics, using novel imaging and microscopy techniques, allows characterization of even full product formulations while validating fundamental models of diffusion and convection in these systems. While some swelling polymers offer ways to build mixing into reconstitution, novel materials and more significant effects are needed and sparse fiber colloids offer one possible approach.

Breakout Session & Reporting Back

The objective of the breakout sessions was to identify the gaps from industry on either understanding the mechanisms involved or the characterization techniques required to adequately wet a powder.

Most of the discussions revolved around three topics:

1. The challenging physics involved in the wetting subprocess:

Capillary forces are difficult to quantify. In addition, the solid does not remain static and heterogeneities start to appear across the powder particle surface due to the wetting process itself, the clogging of the pores and the start of the dissolution process.

The use of long-established equations, such as Washburn are not suitable for these "real particles" and there is a clear need to learn from other fields, such as soil science, to leverage on the learnings on soil imbibition and drainage, including any possible hysteresis between the two processes.

Moreover, the wetting process can also yield the appearance of fish eyes, which are gel-like structures which significantly hinder the powder wetting and can lead to powder swelling and, in the worst cases, impede the total reconstitution of the solid into the liquid phase.

2. Quantifying how easy/fast a powder is wetted:

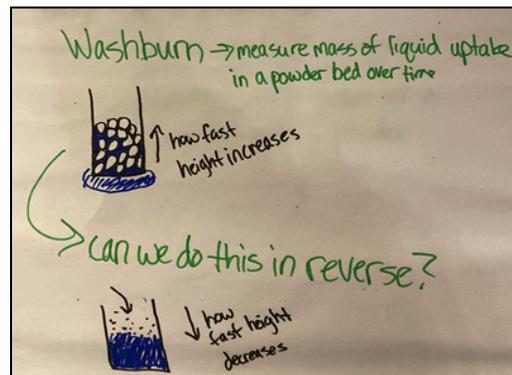
As of today, measuring the contact angle of a drop laid on a powder bed is the principal technique that is widely used to quantify wettability. Nonetheless, the audience agreed that it is not the best technique. Its limitations include scale of

the analysis (often over-simplified) and the interpretation of results, in particular because the situation is out of equilibrium. In most cases, the technique is used as a semi-quantitative metric to compare and differentiate between two powder-liquid systems.

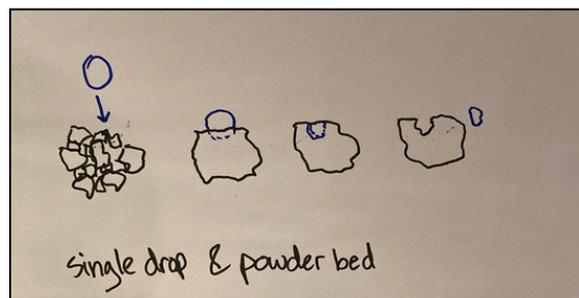
The discussion also led to alternative techniques to assess wettability, for example: What happens when we present a single particle to a liquid surface (a case where equilibrium can be reached)? Which variables can be measured in this case and what can we derive from such experiments?.

Alternative techniques, such as X-ray tomography, liquid pycnometry and porosimetry could also provide some additional hints to better understand the wettability process, linking it to the powder structure properties.

Instead of letting liquid rise through the bed, another suggestion is to conduct an inverted Washburn experiment, in which the powder bed is placed on top of the liquid while the decreasing height is measured (see image below). As an advantage, this technique could allow one to decouple the capillary phenomena (replaced by gravity) from hydrodynamic resistance.



Another idea is a time-resolved measurement in the so-called drop penetration test, whereby a liquid droplet is placed on top of the powder bed while the wetting time is recorded, as shown in the image below:



This simple technique is easily scalable in industry. It may lead to a better understanding of the wetting sub-process by breaking it down into a simpler system.

3. The role of surface properties & wetting liquid on the subprocess:

In addition to points #1 and #2, several properties of the bulk and individual particles challenge our understanding, such as polydispersity (including the presence of fines and potential occlusion of pores) and differences in microporosity.

Other complications rarely considered in modeling include relative humidity of the environment where wetting is taking place, stickiness of the powder, formation of gels and degree of agglomeration.

Moreover, liquid properties (polarity, composition, etc), temperature and flow rate can all impact wetting speed. Therefore, while research efforts should elucidate the behavior of powders in the wetting process, the role of the liquid should also be considered..

The group discussion also revealed limitations of available techniques, and the need to identify key parameters affecting the wetting process, so that proper characterization techniques can be developed.

In conclusion, there is a clear industrial need to clarify the force balance in the wetting process, specifically those acting on the powder while resting on water. The efforts in this field should not be limited to model materials such as hard particles, but should also consider swelling particles and soluble particles should also be considered.

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[2] Alghunaim, A, Techniques for determining contact angle and wettability of powders. Powder technology, 2016. DOI: <https://doi.org/10.1016/j.powtec.2015.10.002>

Workshop Session 3: Spatiotemporal Transformation of Solids During Reconstitution

The third session of the workshop served to identify gaps in our understanding of the transformation process. The reconstitution process spans the combination of liquid and powder, the wetting of the powder, the transformation process, and generation of the final product. As mentioned above, the mechanisms of the reconstitution process includes imbibition, particle swelling, sinking, dispersion, and dissolution. From a researcher's perspective, the kinetics of these mechanisms, how they can be modeled and measured, and understanding the physical manipulation of a solid and liquid suspension is of keen interest.

The transformation process focuses on the dynamics of the system after wetting. Localized, time dependent changes of the system include particle aggregation, viscosity changes and gradients, suspension swelling, and other reactions occurring during the dispersion and dissolution process. In the various applications the amount of energy applied to the system, such as agitation or thermal input, varies. The optimization of the raw materials (particle properties), and the applied energy during the transformation process to prevent in-process failure has been elusive due to poor characterization techniques of these spatiotemporal mechanisms.

The objective of this session was to examine the mechanisms of the transformation process, identify knowledge gaps which would advance the predictability of failure modes observed during transformation, and identify and discuss improvements to the current body of knowledge on characterizing these mechanisms.

Technical Presentations:

Highly Concentrated Particulate Suspensions: Challenges and Opportunities.

Heinrich Jaeger (University of Chicago).

Concentrated, or dense, suspensions of nano- to micrometer-size particles in a liquid can exhibit non-Newtonian flow behavior, where the material's response to shear depends strongly on the intensity of forcing. This talk discussed some of the remarkable, counter-intuitive properties of these materials, focusing on their ability to transform, reversibly, between fluid and solid-like states [1,2]. While earlier work associated the dramatic jumps in viscosity with purely hydrodynamic interactions between particles, more recent experiments and simulations have demonstrated the central importance of frictional contacts [1,3,4]. This opens up new opportunities for designing the dynamic stress response of dense suspensions by tailoring the particles' surface properties. In particular, besides tailoring physical friction through particle shape and surface friction, special opportunities were discussed that arise through chemically functionalizing the particle surfaces with brush layers that can tune the friction [5] and furthermore can introduce highly controllable thixotropic or rheopectic behavior [6,7].

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[3] A. Singh, C. Ness, R. Seto, J. J. de Pablo, and H. M. Jaeger, Shear thickening and jamming of dense suspensions: the roll of friction, Physical Review Letters 124, 248005 (2020).

<http://dx.doi.org/10.1103/PhysRevLett.124.248005>

[4] A. Singh, G. L. Jackson, M. v. d. Naald, J. J. de Pablo, and H. M. Jaeger, Stress-activated constraints in dense suspension rheology, *Physical Review Fluids* 7, 054302 (2022).
<http://dx.doi.org/10.1103/PhysRevFluids.7.054302>

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<http://dx.doi.org/10.1038/s41563-018-0175-5>

[6] G. L. Jackson, J. M. Dennis, N. D. Dolinski, M. van der Naald, H. Kim, C. Eom, S. J. Rowan, and H. M. Jaeger, Designing Stress-Adaptive Dense Suspensions Using Dynamic Covalent Chemistry, *Macromolecules* 55, 6453-6461 (2022). <http://dx.doi.org/10.1021/acs.macromol.2c00603>

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<http://dx.doi.org/10.1039/d3sm00479a>

Rheology of Suspensions Near the Jamming Transition.

Guillaume Ovarlez (LOF, CNRS-Syensqo-University of Bordeaux, France).

The main challenge with suspensions in the industry, when dealing with civil engineering materials such as cement pastes or foodstuffs such as chocolate pastes, is to be able to handle suspensions that are as concentrated as possible. This talk discussed the behavior of concentrated suspensions of non-Brownian particles at high concentrations and its origin. The focus was on the jamming volume fraction, at which the viscosity of such suspensions diverges. The role of interparticle friction, particle shape, and polydispersity on its value was discussed. The current understanding of the rheology of these systems now allows for the proposal of various paths to increase the range of particle concentrations for which suspensions are flowable, either through a change in the mix design or through a change in the flow history.

Chaotic Stokes flow in 3D Granular Porous Media and its Impact on Solute Mixing.

Yves Meheust (University of Rennes).

Solute mixing occurs as soon as the spatial concentration of a solute is not homogeneous within the liquid that carries it. At sufficiently high Péclet number but low Reynolds number, the dynamics of a solute plume evolves under the opposite actions of lamella width compression (induced by longitudinal stretching of the lamella) and diffusive lamella widening. The fluid stretching history controls the dynamics of an advective backbone of the solute plume, upon which diffusive widening can be applied locally to infer the full solute transport and mixing history. Hence, the stretching properties of Stokes flow play a critical role in controlling the mixing dynamics. About ten years ago, numerical and theoretical works showed that Stokes flow in the pore space of continuous porous media is intrinsically chaotic, that is, that the length of material lines grows exponentially in time. We investigated if this property also holds for granular porous media. Using numerical simulations in ordered granular media [1, 2] and experiments in random granular media [3], we demonstrated the exponential

stretching of material lines and measured the associated Lyapunov exponent (i.e., the associated logarithmic growth rate)[1-3,5]. Chaos is generated through transverse intersection between stable and unstable two-dimensional (2D) manifolds, which are separation surfaces for the flow inside the flow domain [1,2]. From this mechanism, we proposed a prediction of the Lyapunov exponent inside packs of monodisperse beads and validated it from both the numerical and experimental data [3]. Due to this exponential stretching, the Batchelor scale, below which the diffusive spreading rate can homogenize solute concentrations against the stretching rate, varies as the inverse square root of the Lyapunov exponent times the Péclet number [3, 4]. Therefore, in most natural and industrial granular porous media of practical interest (pore size ~ 0.1 mm), the solute cannot be well-mixed at the pore scale as soon as the Péclet number is larger than 5 (which is usually the case).

Advective mixing associated with Stokes flow is intrinsically chaotic in all types of three-dimensional (3D) porous media. The mechanism for chaotic advection, and the resulting strength of chaos (e.g. as measured e.g. by the Lyapunov exponent), is controlled by the topology of the flow. The Lyapunov exponent can be predicted theoretically if a characteristic length scale can be defined for the medium. Due to the chaotic nature of 3D Stokes flow, solutes are badly-mixed at the pore scale for most configurations of practical use, which implies that classical continuum scale models of solute mixing are not fully relevant in many such configurations.

Breakout Session & Reporting Back.

This session prompted workshop attendees to discuss the challenges and gaps in predicting failure modes during the transformation process and characterizing the mechanisms. Several challenges were identified across breakout groups, with four common themes mentioned by multiple groups.

1. Reconstituting solids into dense versus dilute systems presents distinct challenges.
2. Reconstitution applications, spanning various length and time scales, result in inherently dynamic and unsteady systems.
3. The ability to identify and measure specific individual particle properties, crucial for tunable reconstitution stages, is currently lacking; and
4. Three-phase systems, involving solids, liquids, and gasses, pose significant rheological challenges

A central theme was the contrasting behaviors observed during reconstitution into dense versus dilute systems. Dense systems, prone to jamming and influenced by shear intensity, pose distinct challenges compared to dilute systems, where inhomogeneous mixing and gel erosion are primary concerns. Addressing the knowledge gap in the "rheological no-man's land" between these extremes was identified as crucial.

Furthermore, the dynamic, multi-scale nature of reconstitution emerged as a significant hurdle. The lack of understanding of individual particle properties and their impact on tunable reconstitution stages was highlighted. The complexities of three-phase rheology, involving air, liquid, and solid interactions, also presented a major obstacle. Participants discussed the need for advanced characterization techniques like microrheology, reliable modeling parameters for simulations, and the potential to leverage knowledge from fields like geoscience. Breakout groups also noted the limited research on reconstitution into non-Newtonian fluids and the challenges of scaling up processes. Ultimately, the session underscored the necessity for interdisciplinary approaches and fundamental studies to advance the predictability of failure modes and improve characterization of the transformation process.

Workshop Session 4: Gap Identification

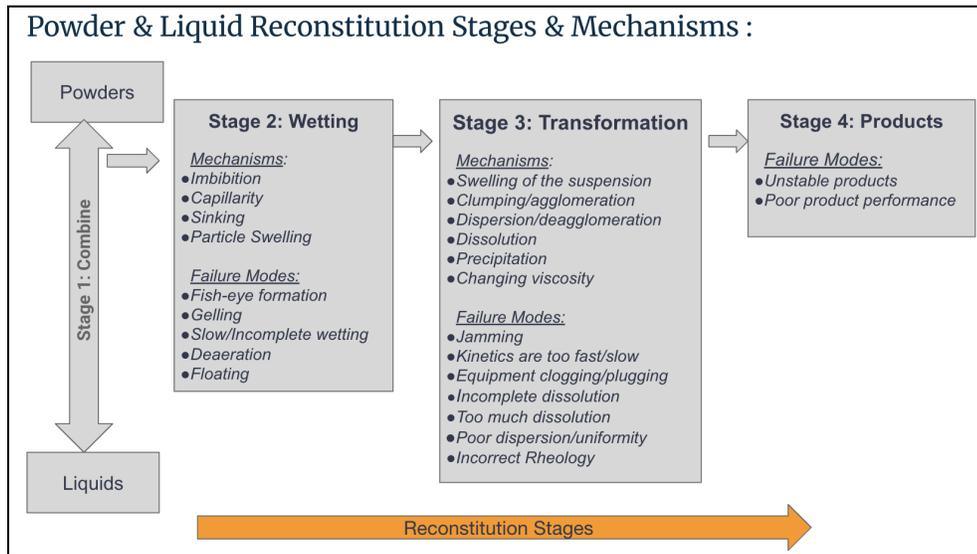
While the first three sessions of the workshop focused on specific use cases and advancements in academic research, Session 4 brought attendees together to identify common gaps and opportunities for industry-wide improvements. This session aimed to initiate the synthesis and prioritization of ideas for future IFPRI research projects.

The session began with participants gathering in breakout groups to summarize the most important concepts from the workshop. Group leaders then presented their teams' key takeaways to the broader workshop audience. The session concluded with brief feedback and suggestions from four academic guests of the workshop, Gul Ozcan-Taskin, Heather Emady, Jeff Morris, and Suzanne Kresta.

Gap 1: Industry lacks Common Language and Relevant Model Systems.

A. Starting framework for powder/liquid reconstitution:

Scientists and engineers often focus on niche applications within powder-liquid reconstitution. However, our community lacks a comprehensive framework capturing the broader, holistic nature of reconstitution processes. The figure below offers a preliminary framework for discussing the key stages, mechanisms, and potential failure modes involved in these systems. While it is not exhaustive, it serves as a valuable starting point for further exploration and development of a unified approach.

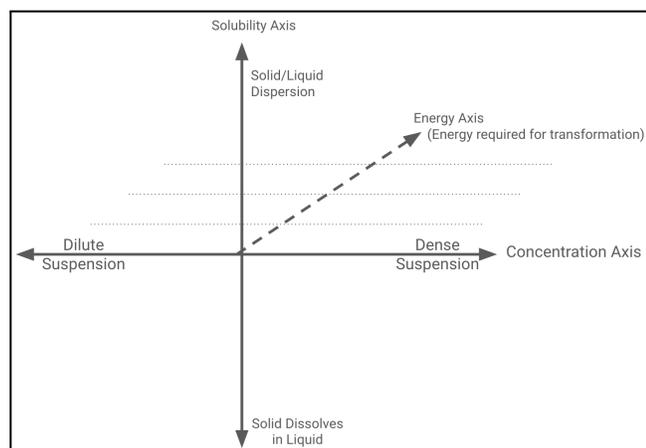


B. Lack of model systems:

To communicate and close gaps in reconstitution effectively, we must first identify model systems for which academic investigators can build meaningful projects. Model systems should be simple, easy to visualize and comprehend. While each model system should cover as wide a range of applications as possible, we recognise that the 'reconstitution space' is too broad to be spanned by any single model system. Instead, we propose to describe this space along three axes, and seek model systems to cover different parts of this space. The three axes are:

- Concentration (dense vs dilute suspension)
- Solubility (dissolution vs dispersion)
- System energy (low to high energy via mixing, shear, heat, etc)

The plot below shows a 3 dimensional visualization of these axes. This is a helpful tool for visualizing and comparing model systems that differ significantly from one another.



One additional variable, time, is also critical to reconstitution processes. The time variable will be addressed later in the report and is most applicable to the complexity and/or kinetics of a single model system. In the above diagram, one may perhaps include time by a system being reconstituted as following a *trajectory* in this space as the process progresses. Proposals for model systems are presented later in this report.

Gap 2: Reconstitution Involves Three Phases.

Industry often underestimates the critical role of gases in powder-liquid reconstitution systems. Gases occupy both the intraparticle pores and the interparticle voids of bulk powders, which can significantly influence the kinetics and outcomes of reconstitution. Complex pore networks, such as those in activated carbons, zeolites, and porous polymers, trap considerable gas volumes. During the wetting stage of reconstitution, liquid must displace these trapped gases within and between particles. This displacement process directly affects wetting efficiency, reconstitution speed, and the stability of the final product. Neglecting the behavior of gases in these systems can lead to inefficiencies and suboptimal performance, underscoring the need for a deeper understanding of their role.

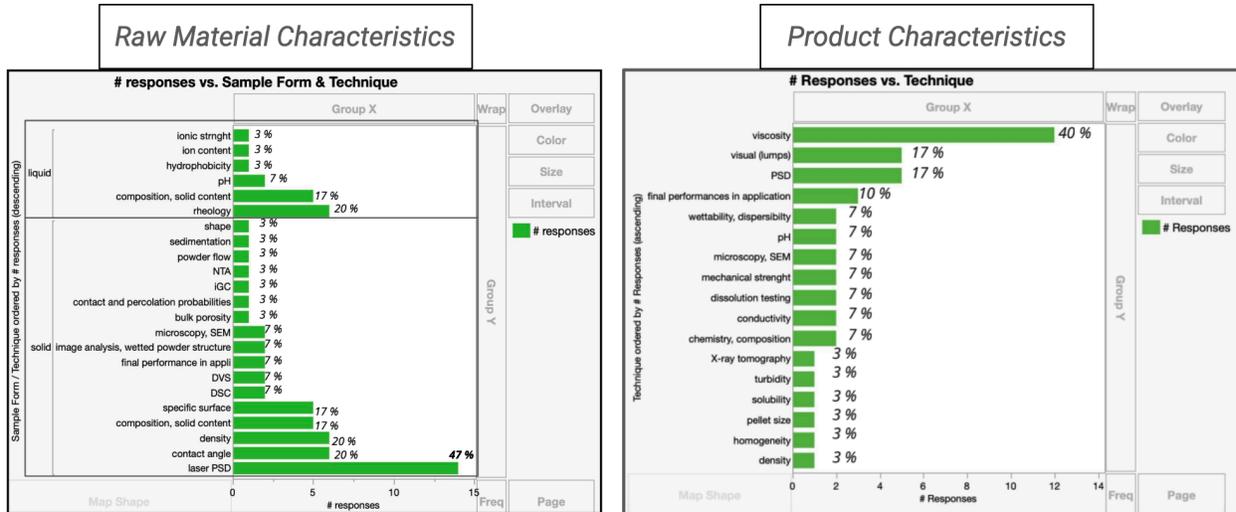
Gap 3. Characterization Gaps in Industry.

The IFPRI member survey revealed significant gaps in characterization practices among members, a finding that was further confirmed through discussions during the workshop. Given the broad scope of characterization, we will address this topic by dividing it into four key components.

1. *Raw Material Characteristics* include properties of the starting liquids and powders. Examples include particle size distribution (solids), surface area (solids), ionic strength (liquid), and pH (liquid).
2. *Intermediate Materials Characteristics* describe properties of materials that have been partially converted or transformed but require further transformation to become the final product.
3. *Process Characteristics* describe the transformation process and are often monitored over time. These measurements are typically conducted in one of three ways: in-line (directly within the system), on-line (using integrated and automated sample collection with real-time measurements), or at-line (involving manual sample collection and immediate analysis after sampling).
4. *Product Characteristics*, which describe the final product in its usable form. These include physical attributes, chemical or compositional properties, and performance tests.

Significant gaps exist in how industry characterizes raw materials and reconstituted products. For instance, fewer than 50% of survey respondents reported using laser diffraction to measure particle size distribution of starting

powders. Even fewer—fewer than 20%—use techniques like contact angle analysis, density measurements, or BET surface area. Moreover, there is little to no consensus on characterizing properties of starting liquids, or even those of the reconstituted products.



Methods of characterization used by IFPRI members showed little consistency, according to a survey completed by 30 members before the workshop.

Effective characterization is essential for successful reconstitution, as it enables a deeper understanding of key properties, interactions, and the kinetics of the system. Identifying critical properties, such as particle size, surface area, porosity, and rheological behavior, is crucial for optimizing processes and ensuring product consistency. Funding research aimed at reducing characterization gaps can significantly benefit industry by providing more accurate, standardized methods, ultimately leading to improved performance, stability, and scalability across a wide range of applications. However, such research must take place alongside a parallel effort to understand the fundamentals of the reconstitution process itself, because without such understanding, we do not know which properties are actually relevant for controlling processes characteristics and final product properties.

Gap 4: Reconstitution Kinetics are Dynamic and Complex.

The rates of reconstitution stages are often interdependent. For example, when hot water is poured over ground coffee, its kinetic energy enhances wetting, which likely accelerates reconstitution. In contrast, adding ground coffee to hot water without agitation results in slower wetting kinetics. The order of operations—whether powder is added to liquid or vice versa—and the energy applied to the system significantly influence the wetting stage. Similarly, the kinetics of coffee extraction, or the transformation stage, depend on both the wetting rate and the sequence of operations. Due to the dynamic nature of this

process, accurately modeling and mathematically describing the kinetics of coffee reconstitution remains challenging.



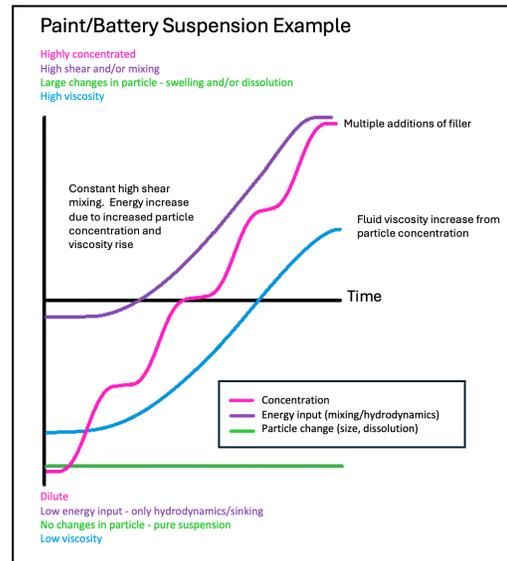
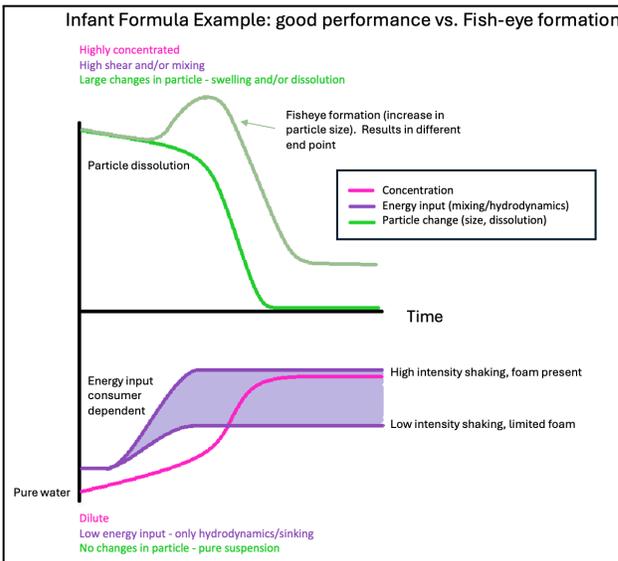
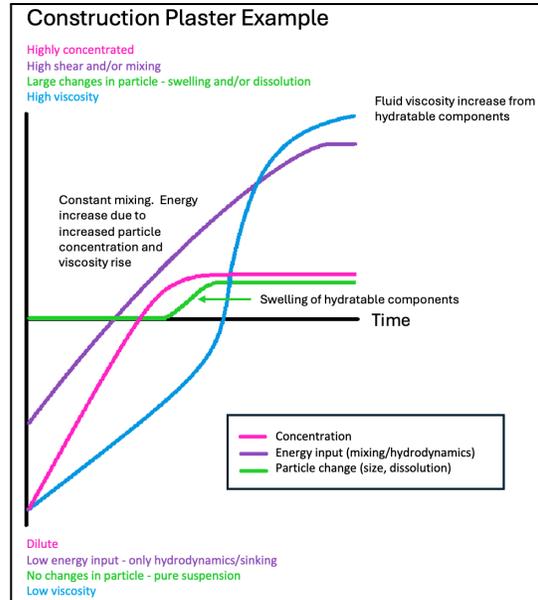
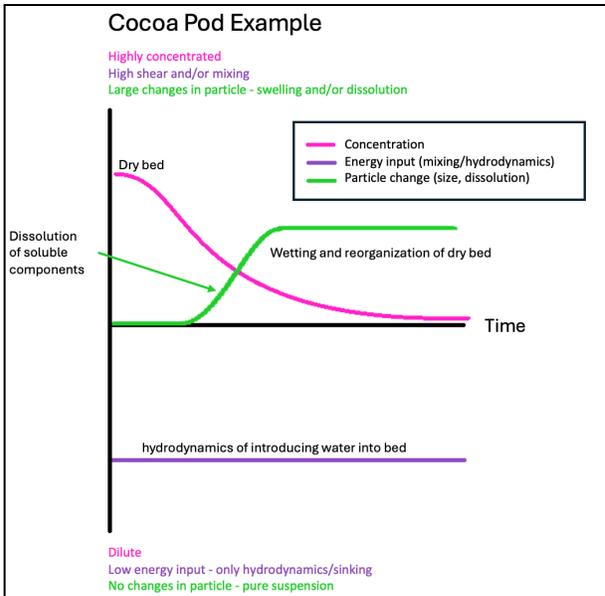
To explore the mechanisms of wetting, we must first define and characterize the system. This can be challenging due to the system's dynamic nature and the various rate-dependent processes at play. However, once the system is properly described, we can analyze how it evolves over time, allowing us to resolve the rates and associated kinetics of reconstitution.

Several key parameters must be considered when describing reconstitution systems. One important factor is **particle concentration**. When liquid is added to a powder bed, the concentration starts high and gradually decreases. Conversely, when powder is introduced into a liquid, the concentration is initially dilute but increases as more particles are incorporated into the suspension.

Another critical parameter is **energy input**. Energy primarily influences mixing intensity and, in many cases, affects solubility through temperature changes.

A third key factor is **particle transformation over time**. Particles that swell or dissolve behave differently than those that change size due to shear forces or remain largely unchanged. In some systems, fluid viscosity also evolves over time, significantly influencing the reconstitution process.

Below are four examples of common wetting challenges encountered in industry. These cases illustrate the diversity of applications and the unique time-dependent behaviors that each system exhibits.



A deeper understanding of these interdependent mechanisms is essential for improving process predictability and efficiency. To overcome existing challenges and drive innovation, targeted research is needed to close knowledge gaps, refine kinetic models, and develop better strategies for industrial applications.

Gap 5: Failure Mechanisms During Technology Scale-up.

One of the most significant—and often frustrating—gaps in industrial reconstitution is the lack of understanding behind failure mechanisms.

Compounding this challenge, many failures do not emerge during research and development, but instead arise during technology scale-up, when the financial and operational risks are much higher. One notable failure mode discussed during the workshop was the formation of fish eyes; clumps of undispersed powder that resist breaking apart in suspension, leading to processing inefficiencies and product inconsistencies. To effectively mitigate this issue, a deeper understanding of the mechanisms driving fish eye formation is essential, including factors such as wetting kinetics, particle surface energy, and mixing dynamics. By identifying the underlying causes, companies can develop targeted strategies to optimize dispersion and improve product performance.

Fish eyes, as well as other failure mechanisms in reconstitution, are not unique to a single industry—sectors ranging from pharmaceuticals and food production to coatings, cosmetics, and battery slurries all struggle with similar challenges. Investing in research to bridge these knowledge gaps will enable companies to solve complex formulation and scale-up challenges more efficiently, ultimately driving innovation and improving processing across industries.

Feedback From Guests:

To close the workshop, some of the invited academic guests were asked to provide feedback, insight, and perspective to consider for future research and collaboration into reconstitution with the IFPRI community. Gul Ozcan-Taskin, Heather Emady, Jeff Morris, and Suzanne Kresta graciously accepted the opportunity to share their insights.

To summarize their feedback, the workshop underscored the complex, multidisciplinary nature of powder reconstitution in liquids, emphasizing the need for a comprehensive approach to understanding and predicting this process. A central theme was the development of robust methods for characterizing wetting and predicting reconstitution, moving beyond the limitations of traditional contact angle measurements. This involves leveraging existing knowledge of particle formation, liquid properties, and flow dynamics to design novel tests that combine material characterization with advanced wetting analysis.

Our guests shared a common belief that the development of well-defined model systems, using agreed-upon materials with varying properties (hard, partially soluble, and fully soluble), was essential for understanding fundamental mechanisms. These systems need to be designed with careful consideration of equipment and scale, recognizing that small-scale techniques, such as sonication, may not translate to large-scale industrial processes. Such considerations ensure that the findings are relevant and applicable to real-world applications. These model systems should be inclusive to characterization of interparticle forces, their impact on rheology and dissolution rates, and the modeling of stress and flow fields. The 'fisheye' and 'lump' phenomena were identified as key areas of study, representing microcosms of the reconstitution problem that require a deeper understanding of local energy requirements and formation mechanisms.

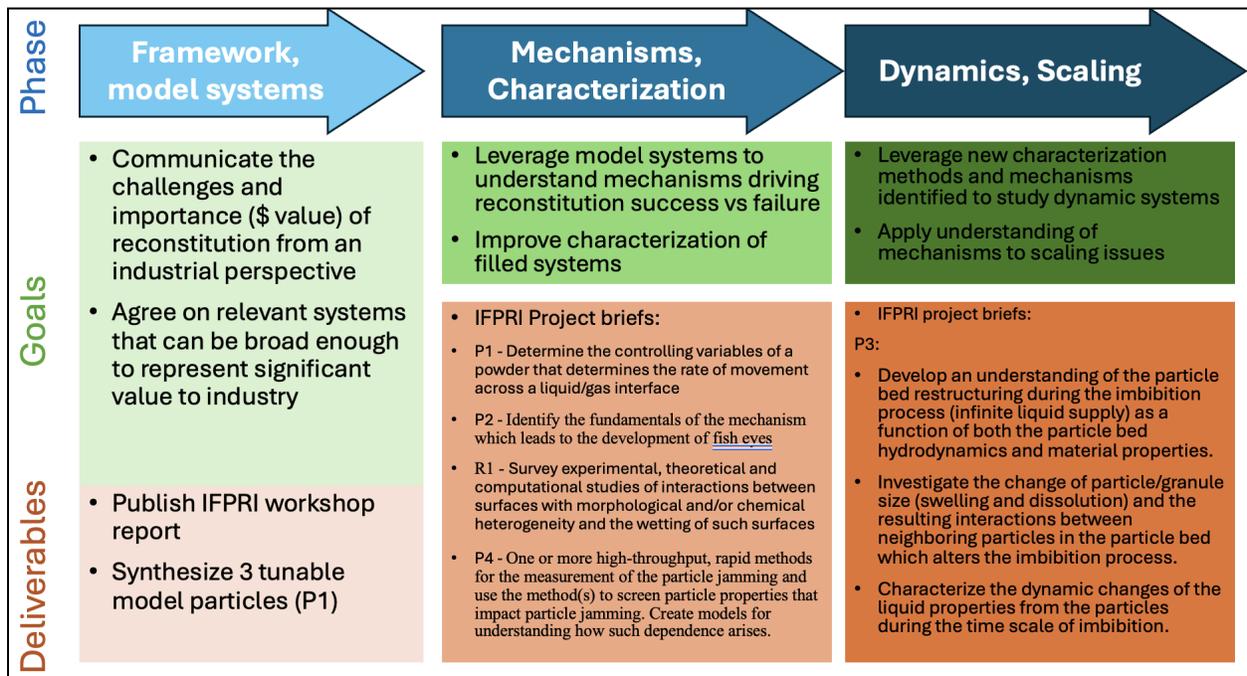
Furthermore, the interplay between turbulent mixing and competing rates in industrial processes was emphasized, along with the importance of understanding the surface properties of heterogeneous powders. The development of regime maps, incorporating different flow fields,

was proposed to quantitatively analyze component interactions and identify undesirable properties. A matrix-based approach, systematically investigating energies and flow field parameters, was suggested to optimize wetting and prevent lump formation. Our guests aligned on a broader long term need is to develop predictive tools and numerical models that can be scaled from laboratory to industrial settings, fostering a deeper understanding of the underlying physics and facilitating efficient and effective reconstitution processes.

Reconstitution Roadmap:

A systematic approach is required to progress the understanding of reconstitution processes. The outcomes of the workshop have been summarized into a roadmap of suggested steps to help advance the field. The roadmap lays a progression to follow for advancing the field of reconstitution and does not include a timeline due to the uncertainty in when work will be funded to achieve the stated milestones. This roadmap should be leveraged annually for the creation of briefs at the IFPRI Annual General Meeting (AGM) and used to track progress towards the higher complexity questions identified in the workshop.

Proposed Roadmap:



Notes & Outcomes from IFPRI 2024 AGM:

One of the objectives of the Annual General Meeting (AGM) is to define, discuss and adapt the content of the technical program. Therefore, a significant portion of the AGM is dedicated to writing briefs that address topics of interest, aiming to define future research projects to be included in the IFPRI program. In 2024, this AGM occurred

immediately after the workshop. This constitutes a great opportunity as discussions that happened during the workshop played a crucial role in feeding the ideation process.

In total, 19 briefs were proposed, 7 of them being specifically related to powder reconstitution topics and discussions that took place during the workshop (4 projects, 2 reviews, and 1 workshop). After final selection, 6 of these briefs were voted on and have been sent to academics to solicit project proposals (see table below). This collaborative effort marks a significant step forward in our research initiatives.

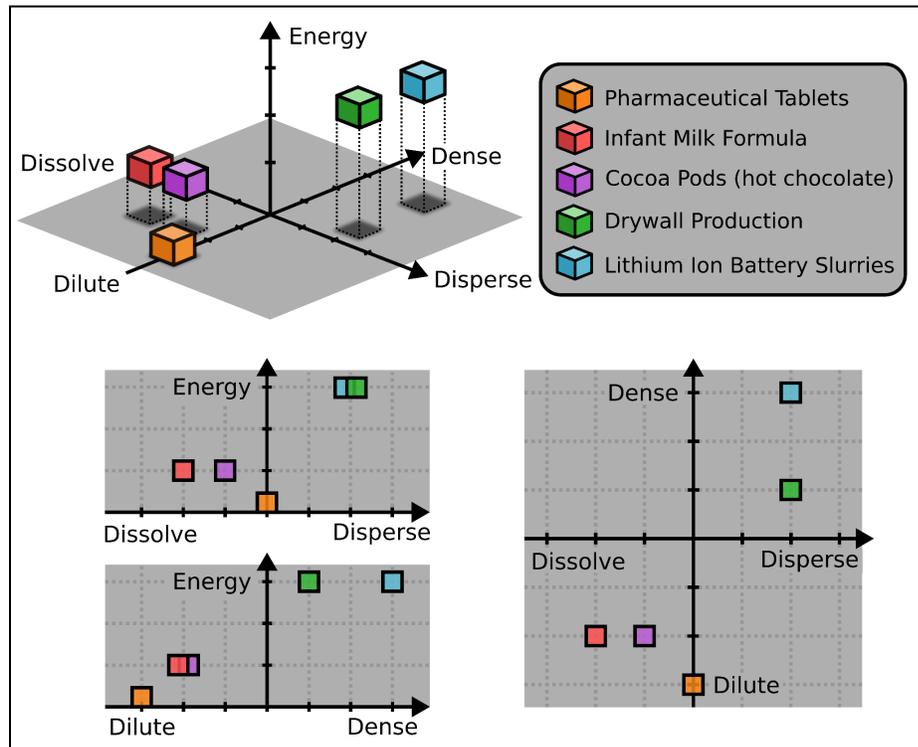
Topics	Ref	Type	IFPRI area	Gaps addressed	Vote results
Surface energy of heterogeneous surfaces	R1	Review	Characterization	Fundamental understanding Characterisation Gaps 1 & 3	Yes
Measurement of multimodal, multicomponent PSD in suspensions	R2	Review	Wet systems Characterization	Tech. methodological & modeling limitations Gaps 3 & 4	Yes (Discontinued during 2025 Winter meeting)
Transport of heterogeneous particles through a gas/liquid interface leveraging tunable model particles	P1	Project	Dry systems Wet systems	Fundamental understanding Gaps 1, 2 & 3	Yes
Fish Eye Avoidance & Break up	P2	Project	Wet systems	Fundamental understanding of mechanisms & modes of failure Gaps 3, 4 & 5	Yes
Dynamic imbibition	P3	Project	Wet systems	Fundamental understanding Gap 4	No
High Throughput Measurement of Jamming	P4	Project	Wet systems Characterization	Tech. methodological & modeling limitations Gaps 3 & 5	Yes
Friction in wet & dry systems	W1	Workshop	Wet systems Dry systems Modeling	Fundamental understanding Characterisation Gaps 1, 3 & 4	Yes (2026)

Proposed Model Systems for Reconstitution:

When describing and differentiating model systems of reconstitution, we must start with simple and common descriptors. At a high level, we believe reconstitution systems can be described using three categories:

1. The **concentration** of solids within a suspension is a crucial factor, influencing its behavior and categorization as either dense or dilute. A suspension is considered dense when it contains 50% or more solids by volume, while a dilute suspension contains less than 50% solids by volume. While these 50% thresholds serve as general guidelines, the precise classification of a suspension as dense or dilute can vary significantly across different systems. This variability arises from factors such as particle size, shape, density, and the suspension's rheological properties. The transition from dilute to concentrated behavior is often more accurately defined by the onset of a measurable yield stress. In well-stabilized, non-adhesive suspensions of spherical or near-spherical particles, a significant yield stress typically emerges abruptly at the frictional jamming point, approximately 55% solids by volume. However, when interparticle forces such as friction and adhesion are present, a yield stress can develop more gradually and at lower solid concentrations, potentially as low as 20%. Rheology, the study of the flow of matter, is the preferred technique to quantify these properties.
2. The **solubility** of solids in the liquid matters. During the wetting and transformation stages, solids are either dispersed to form a suspension or dissolved to yield a solution.
3. The **energy input to the system** matters. Energy may be added to the system during wetting and/or transformation through mixing, pouring, pumping, heating, the application of shear, and other sources. System energy can span a wide range of low to high energy.

Systems become more complex on a case by case basis. We recommend starting by describing model systems by their concentration (dense or dilute), solubility (dissolve or disperse), and by the system energy used during the transformation (low, medium, high energy or shear). This serves to define boundaries and narrow the scope for reconstitution research projects. We provide examples of industrially relevant reconstitution types below, which include lithium ion battery slurries, pharmaceutical systems, infant milk formula, construction plaster, and cocoa pods.



Lithium Ion Battery Slurries:

Slurries made during the manufacturing of lithium ion batteries serve as an excellent example for a model system of powder and liquid reconstitution. Battery slurries are a mix of powders and liquids that are combined with high shear to make a dense and viscous suspension (powder is dispersed, not dissolved). This slurry is then coated onto a thin sheet of foil and dried to form an electrode (anode or cathode). The paragraphs below, prepared by the Centre of Industrial Rheology, provide an excellent overview of the battery slurry reconstitution process ¹.

“Electrode slurries are a mix of solid, conductive particles along with active materials, polymer binders and a solvent medium. The performance of the cell is highly dependent upon the electrochemical properties of the active materials as well as the distribution of the components within the electrode slurry, the connectivity between the components and the adhesion of the electrode with the current collector.

Generally speaking, the slurry is prepared by mixing any solid components, wetting the mixture with an appropriate solvent and finally forming the suspension, which has to be degassed before use to remove gas voids and avoid coating defects. Once prepared, the slurry film is applied to the substrate and dried by evaporation. During mixing of the solids, it is widely accepted that deagglomeration of carbon black leads to more homogenous mixing and increased carbon black adsorption onto the surface of the active material. Wetting the solid mixture by adding solvent in differing quantities can

lead to different states of adhesion and stress, as well as agglomeration of solid particles; these properties can be monitored using powder flow rheology.

Changing the wetting solvent and diluting to different concentrations can lead to significant rheological differences in slurry behavior. For example, having a highly viscous medium for an electrode slurry can impart many benefits to stability, storage and application. It has been noted that gradual dilution of a dense slurry can give a better dispersion state and battery performance, but excessive dispersion can lead to a deterioration in performance. This is suggested to be due to destruction of electrical conductivity paths.”

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Pharmaceutical Systems (Tablets)

The reconstitution of solid powders in pharmaceutical systems includes different applications.

In pharmaceutical technology reconstitution takes place during manufacturing intermediates and products, e.g. by the addition of powder (and air) to liquid for the preparation of suspensions with a subsequent removal of the liquid and deposition of particles during spray-layering and film-coating processing or separation of particles during spray-drying processing. The particles may consist of insoluble active pharmaceutical ingredients and/or excipients with the potential addition of soluble polymers and surfactants for functionality reasons.

Another application in pharmaceutical technology during manufacturing intermediates and products includes the addition of powder (and air) to liquid for the preparation of solutions with a subsequent removal of the liquid and deposition of polymers and/or surfactants during spray-granulation and wet-granulation processing or separation of particles during spray-drying processing. The particles in the latter case may consist of soluble active pharmaceutical ingredients and soluble polymers for functionality reasons.

The reconstitution by the addition of powder (and air) in liquid for applications in pharmaceutical technology is typically performed by the simple, gravimetric addition of powders onto the surface of slowly stirred liquids. Typical challenges during these reconstitution processes are poor wettability of insoluble active pharmaceutical ingredient, sometimes in combination with no/almost no deaeration leading to floating of the powder onto the liquids’ surface or to agglomerates and clumps in the liquid. In special cases high shear mixers are applied to facilitate dispersion into primary particles and to promote deaeration of air located within the powder.

Another application of reconstitution of solid powders in pharmaceutical systems is related to the administration of solid dosage forms [1]. This includes in particular orally administered tablets and capsules. Tablets are compacted powders with about 10-20% porosity. Hard-shell capsules are most often made from gelatin or hydroxypropyl methylcellulose, and the almost no or slightly compressed powder is filled inside the capsule. The reconstitution takes place upon administration in the gastrointestinal tract. The composition of the liquid, amount of liquid and fluid- and mechanical-energy for reconstitution is strongly varying in the gastrointestinal tract [2,

3]. Tablets (consisting of the powder) and capsules (powder inside the capsule shell) consist of soluble active pharmaceutical ingredients and insoluble and soluble excipients for filling purposes and functional reasons. Typical challenges during these reconstitution processes are poor wettability of the soluble active pharmaceutical ingredient, negative impact of liquids' pH and ion composition/strengths on the solubilization capacity of the active pharmaceutical ingredient, small amount of liquid and small hydrodynamic forces with the negative implication of poor or highly variable absorption of the active pharmaceutical ingredient. It must be highlighted that these processes are highly complex and depend on dynamic behavior and are up-to-date and poorly understood. These challenges must be addressed by the formulation and the characteristics of the individual components and the drug product, finally aiming for robust functional behavior including disintegration and dispersion upon administration under varying conditions in the gastrointestinal tract.

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Infant Milk Formula

Powdered infant milk formula is a manufactured food designed to feed babies and infants. It is typically made from modified cow's milk protein to resemble breast milk, containing essential nutrients such as lactose, vegetable oils, vitamins, and minerals [1].

The formula is mixed with water or another liquid before feeding. There are various types of infant formulas, including those made from cow's milk, goat's milk, soy protein, or hydrolyzed proteins. Thus, it is a complex system given the large number of ingredients present in the powder, each of them with their own physical and chemical characteristics.

Reconstituting powdered infant formula can present several challenges, primarily due to the need for precise mixing conditions and hygiene standards. One common issue is the formation of clumps, which can occur if the powder is not adequately mixed with water at the correct temperature (2). Additionally, the presence of certain ingredients, such as stabilizers, may require higher shear forces to dissolve properly (3).

Problems with reconstitution can happen both at the factory, during ingredient dissolving (normally delivered in a powder format) or at consumer's homes. At an industrial scale, high shear mixers, temperature control and other mechanisms are available to promote a proper reconstitution. At parents homes, the situation is more complex, limited shear is available, coming only from shaking or swirling the bottle around, so the powder must be engineered in such a way that it dissolves fast without leaving lumps behind.

Improper powder reconstitution is one of the most common complaints from consumers, thus, addressing these challenges is key for the Infant Formula manufacturers.

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Construction Plaster

Construction plaster is mainly used in the creation of drywall, drywall finishing products, and floor screeds. When plaster is reconstituted with water it chemically binds with water to create gypsum. This inorganic material is used in the building industry because of its fire resistance properties, due to the chemically bound water, and its abundance around the world. Furthermore, while plaster is being made from dehydrating gypsum, the particle shape is manipulated from needles to cubic structures based on processing.

Based on the production speed and product performance, drywall, drywall finishing products, and floor screeds are all reconstituted using different plaster particle shapes, additives, and mixing types, each with its own unique challenges. Drywall production requires higher water to solid ratios, very quick high shear mixing, low water demand additives and high fluidity to shape the slurry into drywall very rapidly. Drywall finishing products are designed to have lower water to solid ratios, mixed by the customer and have water absorbing additives for hand spread ability. Floor screeds require very low water to solid ratios, less than 1 minute mix times, medium shear mixing, and have additives which level the poured slurry.

Although each of these products is made daily and in high volume, faster and more complete reconstitution is always a goal to increase capacity and reduce defects. Complete wetting of the plaster is critical because the hydration reaction from plaster to gypsum is what fundamentally characterizes the product. However, the free water that binds to the plaster to create gypsum is no longer available for neighboring particles to use for flowability or for hydration. Additionally, when products have water absorbing additives a competition exists for free water. Incomplete wetting, reduced hydration, and lack of water create issues such as reduced strength, lumps, fish eyes, etc.

Understanding the basics of wetting in these systems will enable progress towards faster and more complete reconstitution.

Cocoa Pods

Single serve powdered mixes and single serve beverage machines have disrupted the way consumers prepare beverages at home or in the workplace.¹ One category of beverages within the single serve beverage market is reconstituted indulgent beverages. Indulgent beverages include hot cocoas, lattes, and cappuccinos amongst many others. These beverages are often comprised of milk powders (dried whole milk, cream, or nonfat dried milk), sugar (powder and/or granulated), salt, cocoa, soluble coffee, flavors, gums, colors, silicon dioxide, and sucralose.^{2,3,4}

Each of these ingredients have various physical and chemical properties adding to the complexity of the reconstitution process for a product containing a blend of ingredients. As an example, the particle size varies greatly amongst these materials spanning nearly two orders of magnitude, ranging from several hundred microns (e.g. granulated sugar ~200-500 μ m)⁵ to single and tens of microns (e.g. 10x powdered sugar and spray dried dairy powders).^{6,7} These blends, containing wide ranges of particle size, are strong candidates for various granulation techniques. Granulation processes such as steam-jet agglomeration, fluid bed granulation, high shear granulation, and agglomeration within a spray drier are often employed to address segregation, reconstitution properties, and improve powder flow.^{8,9}

The composition of individual ingredients, such as milk powder, plays a large role in the reconstitution process. Whole milk contains approximately 30% fat, 27% protein and 38% lactose on a dry basis, whereas non-fat dried milk contains 52% lactose, 39% protein, and 1% fat on a dry basis.¹⁰ Reconstitution time of milk powders was reported to be dependent upon protein composition and particle structure, i.e. agglomerated versus non-agglomerated.⁹ Agglomerated whey protein powder resulted in faster wetting times reducing overall reconstitution time, whereas agglomerated casein protein powder had a slower reconstitution time.^{11,12} The age of the dairy powder and the temperature and humidity in which it is stored can affect the reconstitution time as the powder undergoes physicochemical changes. These changes can include lactose crystallization, free fat mobility to the particle surface, and destabilization of milk protein—all of which affect various stages of the reconstitution process.¹³

It is apparent how the composition of these powdered beverages with broad physical and chemical properties, and various sensitivities to environmental storage conditions can pose challenges in reconstitution. These challenges are observed in agitated vessels where more flexibility exists on impeller speed, liquid temperature and time in the vessel.¹⁴ These challenges are likely only exacerbated in a system where the volume is constrained, there is little to no agitation provided, and the time for reconstitution is limited to under 45 seconds.¹⁵ There is a gap in understanding of the various failure modes during powder reconstitution in systems where liquid is incorporated into a powder bed with minimal agitation, and where the product undergoes a transformation from a dense system to a more dilute system. Given the prolific manufacturing and consumption of single serve beverage capsules and sachets,

in the tens of billions annually, addressing the gaps in powdered beverage reconstitution phenomena is of critical importance.

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Workshop Summary:

Workshop discussions reinforced a well-known challenge; reconstitution behavior is influenced by numerous variables, yet there is little consensus on which are most relevant or how they interact. Despite a wealth of experimental data, studies rarely build systematically upon one another, making it difficult to develop industrially relevant models. This disconnect between experiments and needs of industrial modeling has led to fragmented progress and poorly defined research questions, potentially deterring academic engagement.

A more structured approach, like Karen Hapgood's agglomerate breakup study, could address this gap. Her work used well-characterized model agglomerates to systematically vary parameters, later revealing flaws in DEM simulations. Similarly, past IFPRI-funded research on suspension rheology leveraged well-defined colloidal systems to produce a reliable body of data, driving significant advances in both experiments and modeling.

Applying this strategy to reconstitution, IFPRI should solicit proposals for model systems that, while not covering all reconstitution processes, allow systematic tuning of key parameters such as wettability and dispersion. Collaborating with industry members, these models can clarify which parameters matter, rule out irrelevant ones, and uncover new factors. A second phase would then build on these findings with targeted experiments and modeling.

Given its cross-cutting nature, this initiative would require coordinated efforts across IFPRI's wet, dry, and characterization programs.

Appendix:

Links to workshop logistics and planning:

- Powder/Liquid Reconstitution [Workshop Agenda](#)
- [Workshop Overview and IFPRI Reconstitution Survey Results](#)

Links to Technical Presentations from IFPRI Industrial Members:

- Brian Levy-Polis, FMC. [Crop Protection Water Dispersible Granules – Formulation Development and Launch Troubleshooting](#).
- Laurent Forny, Nestlé. Science of Reconstitution: Recent Progresses and Upcoming Challenges. *The link to these slides is not available due to privacy preferences.*
- Martijn van der Hoeven. [Challenges in Reconstitution of Infant Milk: What Do Customers Do And How to Measure It?](#)

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- Marty Murtagh, Corning. [The Reconstitution Enigma of Nonmetallic Slurries.](#)
 - Mingzhe Yu, Johnson Matthey. [Manufacturing Dense Inorganic Solids Suspensions: Industrial Challenge Case Study.](#)

Links to Technical Presentations by Keynote Speakers:

- Mathis Fricke (TU Darmstadt). [Capillary Imbibition: Progress in Understanding Powder Wetting Phenomena and Applications.](#)
- David Quere (ESPCI Paris). [Grains at Interfaces - Principles and Applications to Liquid Marbles.](#)
- Pat Spicer (University of New South Wales). [Sparse Fiber Structures to Minimize Powder Mass and Maximize Rehydration and Mixing.](#)
- Heinrich Jaeger (University of Chicago). [Highly Concentrated Particulate Suspensions: Challenges and Opportunities.](#)
- Guillaume Ovarlez (LOF, CNRS-Syensqo-University of Bordeaux). [Rheology of Suspensions Near the Jamming Transition.](#)
- Yves Meheust (University of Rennes). [Chaotic Stokes flow in 3D Granular Porous Media and its Impact on Solute Mixing.](#)

Breakout Group presentations, slides, and notes from all 4 workshop sessions:

- [Group 1](#) Breakout Sessions & Reporting Back Notes
- [Group 2](#) Breakout Sessions & Reporting Back Notes
- [Group 3](#) Breakout Sessions & Reporting Back Notes
- [Group 4](#) Breakout Sessions & Reporting Back Notes
- [Group 5](#) Breakout Sessions & Reporting Back Notes
- [Link to Notes Taken by Facilitators of Workshop Sessions](#)