Proposal to IFPRI members for a PhD project on

POWDER RECONSTITUTION

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Introduction/ Problem definition

From food, personal care, pharmaceutical products to energy applications/batteries, paints, lubricants, coatings, wastewater treatment, minerals processing, there are ample examples in industry which require incorporating particles into liquids. Due to the wide range of applications, physical properties of the materials and final products vary considerably, dictating process targets.

Density differences result in the particles sinking or floating, requiring off-bottom suspension or drawdown from the surface and some progress has been made on these: correlation to estimate suspension speed, recommendations on design are available in published research (Özcan-Taşkin et al, 2001; 2003; 2006). With powders, in addition to density differences, other physical properties come to play a role and even particles which are heavier than the liquid phase can float at the liquid surface for long periods, also forming "fisheye" type structures. This is due to (Etchells, 2001)

- poor wettability and/or
- low apparent bulk density resulting from the formation of fisheyes, which are essentially clusters of particles with air entrapped giving an apparently low bulk density.

The formation of such macroscale clusters, containing air and dry powder, depends on both the formulation and hydrodynamic conditions. Once formed, they may maintain their identity for long periods, resisting reconstitution and the generation of a homogenous, fine dispersion.

Improvements can be achieved through modifications of the formulation to affect the different stages of wetting- adhesion, immersion and spreading. With a given formulation though process results can vary vastly as has been shown in previous work (Xie et al, 2007; Özcan-Taşkin, 2015 a; 2015 b). This is due to differences in practice- either in design or operation- resulting in different hydrodynamic conditions, in particular, close to the liquid surface where the powder addition is made. Consequently, even with a given formulation, reconstitution may be fast or significantly delayed with fisheye formations at different sites or from lab to large scale.

The proposed project focusses on process design, operation and scale up as formulations are highly system specific and of confidential nature; where possible generic formulations or part formulations of interest to members will be included.

Powder additions are made to achieve target product properties: the powder may be an active ingredient in the formulation or an additive to ascertain a particular rheological profile, for example, to facilitate the application/use of the final product. Typically, a mechanically stirred tank is used (Figure 1) and the addition is made at the liquid surface. When there is inherent resistance to incorporation, the powder remains at the liquid surface, characterised by low liquid velocities, for long periods. This facilitates the formation of fisheyes which can maintain their structure even after being drawn into the liquid. Such fisheye formations of different sizes in different parts of the bulk result in significant spatial variations in product properties, with adverse effects: cosmetic or functional defects, poorer product performance.

Whilst fisheye formations can be easily dealt with at formulation scale, they pose significant issues beyond lab scale volumes. Hence, success in reconstitution, i.e. success in consistent product properties- within a given batch, from batch to batch, at different sites where different equipment used can be different and upon scale up- is success to commercialisation.

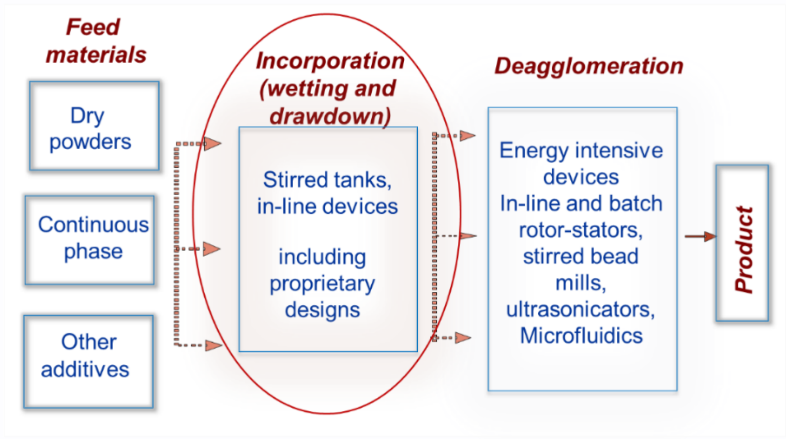

Figure 1. Powder dispersion in liquids (Ozcan-Taskin, 2015).

The focus of this PhD project proposed for IFPRI members following on from discussions during June 2024 meetings and subsequent correspondence is powder incorporation for reconstitution. It will build on the previous work by NGOT with the overall aim of providing recommendations and guidelines for industrial practice for reconstitution where traditional set-up's have severe limitations in preventing the formation of fisheyes.

The main questions to address here are

- How do we take into account material properties for an initial conceptual design that would avoid the formation of "fisheye" structures?
- What approach should be taken to identify the operating window to achieve fast incorporation with different powders and/or powder concentrations without the formation of fisheyes?
- How can the incorporation process be scaled up for large volume manufacture?

Other pertinent questions include

- How to minimise variations during manufacture: within a batch, batch-to-batch including manufacture at different sites with different equipment/configurations
- How can the design and operation be robust and stable during reconstitution to minimise air entrainment from head space, mechanical instabilities, fouling on vessel internals, raise of dust?
- How would the operating window change with evolving dispersion rheology due to powder addition?

Proposed workprogram

The workprogram will make use of a batch and an in-line mixing configurations allowing the introduction of the powder directly into regions of high liquid velocities, turbulence energy dissipation rates or in more viscous liquids into regions where extensional and shear stresses dominate. It is organised in workpackages (WPs) with specific objectives and tasks for each, as described in the Sections below.

WP1- Test case definition and characterisation

The objectives of this WP are to

- Define selected test cases, i.e. representative particle-liquid pairs
- Determine relevant characteristics of particles, liquids and dispersions

Task 1.1 Selection of test systems
The tendency of powders to form clusters is highly dependent on the specific formulation, i.e. not only the particle-liquid pair, but also the presence of other additives in the dispersion. The product also dictates how the formulation may be modified, i.e. there may be limitations in terms of pH adjustment or choice of dispersing agents. Whilst the formulation is often confidential, it should be possible to identify relevant test cases- partial formulations or generic formulations of interest to IFPRI members- to use in this project.
At the start of the project, NGOT will propose powder-liquid pairs with different properties to discuss with consortium members and invite suggestions.
Both the powders and liquid will need to be commercially available, i.e. with MSDS and it may well be possible to source these from an IFPRI member. They will be used in targeted studies: for example a given particle with different surface modification/hydrophobic properties or a given particle manufactured through different routes.
The discussions will also include the liquid(s) to cover the range of interest of continuous phase and dispersion rheology.

Task 1.2 Characterisation of test systems
There are well-established protocols in the research group to characterise of dispersion properties, including particle sizing and rheology. Links with other related activities within IFPRI will also be formed in relation to these.
It is proposed to evaluate the wettability of particles using a static test method, i.e. with no agitation.
The project will also have access to Loughborough University Materials Characterisation Centre (LMCC) which is within the School of AACME (AeroAuto, Chemical and Materials Engineering) where the examination morphology proves to be a useful tool.

WP2- Powder incorporation in batch operation

This part of the study will be performed with a specific batch set up, the inherent design of which allows the introduction of the powder directly into regions of high shear. The proposed study within this WP has the following objectives

(i) Determine the operating window over which powder incorporation can be achieved through a study of the flow characteristics, specifically the suction performance;
(ii) Determine reconstitution kinetics within the above operating window by linking the suction performance to the rate of powder incorporation, also taking into account the evolving rheology due to powder additions;
(iii) Based on reconstitution kinetics, establish the comparative performance of traditional (impeller driven, with surface additions) designs and through introduction to high shear zones: for selected powder-liquid pairs demonstrate what advantages can be gained in avoiding the formation of “fisheye” formation through rapid incorporation
(iv) Provide guidelines on the design and scale up of batch reconstitution processes with minimal risks of “fisheye” formation.

The proposed study will make use of a batch set-up, Ytron Y Jet, building up on the previous work of NGOT. It will be performed at around 20 and 150 l scales, i.e. with tank diameters of around T= 0.30 and 0.60 m. At least at one scale, apart from rapid reconstitution which minimises or eliminates risks of fisheye formation, additional advantages such as minimal dust in the environment will also be covered as part of the program.
It is proposed to use one to two particle-liquid pairs, as identified to be of interest to IFPRI members from within WP1.

Task 2.1 Determination of flow characteristics/ suction performance
Using liquids that mimic the rheological properties of the continuous phase(s) and dispersions the effective operating window will be determined based on suction at the powder inlet.
This part of the project will build on the existing data from the research group.
This Task will be performed at pilot to large scale with existing rigs in the research lab that can handle 20, 100 litres or more.

Task 2.2 Reconstitution kinetics
The Task, which relates to objectives (ii) and (iii), will be performed over the operating window determined in Task 2.1 to link the suction characteristics to reconstitution rate.
Comparisons with a traditional set up will demonstrate the advantages in eliminating fisheye formation. It is proposed to perform the comparative study under selected conditions at a scale of 20 litres, T= 0.30 m. Majority of the study will be performed at T= 0.60 m scale working towards the objective of providing recommendations for design and scale up.

WP3- Powder incorporation in recirculation mode

This part of the study will be performed with a proprietary design in-line rotor-stator, Ytron ZC1, which allows the introduction of the powder directly into the mixer head. The proposed study within this WP has the following objectives

(i) Determine the operating window over which powder incorporation can be achieved through a study of the flow characteristics, specifically the suction performance;
(ii) Determine reconstitution kinetics within the above operating window by linking the suction performance to the rate of powder incorporation, also taking into account the evolving rheology due to powder additions;
(iii) Based on reconstitution kinetics, establish the comparative performance YtronZC1 for selected powder-liquid pairs and demonstrate how “fisheye” formation can be minimised through the appropriate choice of operating conditions to achieve rapid incorporation
(iv) Provide guidelines on the design and scale up of batch reconstitution processes with minimal risks of “fisheye” formation.

The proposed study with Ytron ZC1 will build up on the previous work of NGOT. It will be performed at around 20 and 150 l scales, i.e. with tank diameters of around T= 0.30 and 0.60 m. At least at one scale, apart from rapid reconstitution which minimises or eliminates risks of fisheye formation, additional advantages such as minimal dust in the environment will also be covered as part of the program.
It is proposed to use one to two particle-liquid pairs, as identified to be of interest to IFPRI members from within WP1.

Task 3.1 Determination of operating window based on suction performance
Liquids that mimic the rheological properties of the continuous phase(s) and dispersions will be used to establish the effective operating window through pressure measurements at the powder inlet.
This part of the project will build on the existing data from the research group.
This Task will be performed at pilot to large scale with existing rigs in the research lab that can handle 20, 100 litres or more.

Task 3.2 Reconstitution kinetics
This Task, which relates to objectives (ii) and (iii), will be performed to demonstrate how the suction performance can be linked to reconstitution rate over the effective operating window thereby providing recommendations for design. The effects of operating conditions, namely the pressure at the powder inlet, dictated by both the liquid flow rate and rotor speed as well as dispersion rheology will be shown.

The comparative study will include the smaller scale of 20 litres. Majority of the study will be performed at around 150 litres.

Task 3.3 Hybrid design for difficult-to-wet systems
If any of the test systems of interest to the consortium pose particular challenges in terms of wetting, selected runs will aim to demonstrate the potential to use the equipment as a hybrid set up to avoid fisheye formation through simultaneous incorporation and deagglomertion.

Task 3.4 Design and scale up
Based on the outcome of the experimental program, recommendations and guidelines on design and scale up will be presented to avoid fisheye formation during reconstitution.

Time scale of activities:
The following broadly outline the proposed progress by the PhD student; the emphasis on the different aspects of the project will be discussed with IFPRI members and activities/timescales will be adjusted accordingly:
M1- 6: perform a literature review, complete HSE Forms, training on different techniques, oversee any rig modifications if required, start on characterisation in relation to WP1
Literature review and preliminary characterisation results will be presented to IFPRI members
M7- 19: Reconstitution in batch set up
Recommendations on process design for powder incorporation into liquids
M17- 30 : Reconstitution using a recirculating set up
M30- 36: Final data analysis and write up
Report on how to monitor the process and based on the outcome, how to design the process at different scales of operation.

The project will be supported by other research student projects, including MSc and undergraduate research students, who will work alongside the PhD student. Where possible, i.e. if confidentiality does not prevent, there may well be synergies with other funded projects.

The project will benefit from the following **potential input** from IFPRI members:

- discussions and feedback during meetings with both members and other active academic organisations
- provision of materials/powders, if possible
- short stay of the PhD student at a member organisation site to observe and where appropriate take active part in research trials

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