Project proposal

FISFine

Multiscale Investigation of Fluidization-Induced Segregation in Fine Powders: From Lab Testing to Process Modeling

Research context

Fine powders are essential ingredients in the industrial processing of pharmaceuticals, chemicals, food, pigments, catalysts, and advanced materials. Their high specific surface area enhances reactivity, dissolution and mixing. However, it also leads to strong interparticle forces, such as van der Waals, capillary and electrostatic forces, which dominate as particle size decreases. These forces can severely impair flowability. Because homogeneity is often crucial for product quality, segregation issues at different stages can severely affect performance and economics. Segregation influences the homogeneity of fine powder mixtures during the filling and discharge of storage containers, where fluidization plays a significant role. However, the extent and dynamics of segregation across the scales are still not well understood. Indeed, the effects of segregation often amplify with scale. Further research is necessary to quantify the dominant mechanisms at the fundamental level and link them to industrial processing conditions. This research requires not only specific data from dedicated equipment, but also detailed modeling of the mechanisms acting at the microscopic, particle-scale level to gain multiscale understanding and find potential new mitigation solutions.

State-of-the-art and objectives

During container filling or discharge of fine powders, being it in stagnant or counterflowing air, segregation due to size, density, shape can be observed. The air serves as a fluidizing agent, promoting particle local suspension and potentially segregation. Cohesive forces are very important compared to gravity, rendering the process highly complex to predict, scale-up and control. In fluidization engineering, various decades ago Geldart demonstrated that fine, cohesive powders (Group C) exhibit poor fluidization due to agglomerate formation, and many studies confirm that micro- and nano-sized powders behave as agglomerate clusters rather than individual particles (Kamphorst et al., 2022). To improve handling, binary or multicomponent mixtures are often used, where coarse particles stabilize fine ones and enhance flow, fluidization or dosing performance (Zafar et al., 2017). Frequently, agitating powders with different sizes, densities or shapes with the intent to mix them can actually induce segregation. This issue is increasingly highlighted in recent pharmaceutical studies on blend uniformity and continuous manufacturing, where segregation during transfer or feeding is a major cause of content variability (Schneider et al., 2024).

Fluidization-induced segregation is particularly critical: differences in particle velocities, gas–solid drag and bubbling patterns generate persistent vertical and radial composition gradients. CFD–DEM and experimental works show distinct regimes of mixing and segregation depending on gas velocity, particle size ratios and bed expansion (Di Renzo et al., 2008). A macroscopic model for predicting the segregation direction and equilibrium in binary fluidized bed is the Particle Segregation Model (PSM, Di Maio et al., 2012). A successful attempt to apply physical model to segregating cohesive (wet) material was conducted by Li and McCarthy (2005).

Advanced studies involving PEPT, MRI and high-speed imaging also reveal that small hydrodynamic instabilities can produce large segregation effects in industrial-scale beds (Müller et al., 2008). Despite the progress, large-scale segregation remains difficult to predict. Recent reviews emphasise that empirical correlations often fail at scale, and that DEM-CFD models still require improved closure laws and calibration strategies (Yang et al., 2023; Hadi, 2024). New theoretical work aims to generalise segregation velocity models across flow geometries (Duan et al., 2025).

The FISFine project aims at addressing the following challenges:

1) gaining understanding on the root causes of fluidization-induced segregation during container filling/emptying, particularly in relation to the key material properties;
2) quantifying segregation effects across the scales, by combining experimental and modeling data to link material, process, and operation properties to the likelihood and extent of fluidization segregation;
3) study local conditions around critical container elements (e.g. valves) or openings (e.g. discharge/venting) and propose and evaluate practical mitigation strategies.

The above ambitious objectives will be pursued by a multi-instrumental methodology combining: standard and innovative characterization methods for fine cohesive powders and blends; experimental analysis on lab-scale fluidization rigs capable of quantifying segregation; design and set-up of a dedicated lab-scale facility for container filling/discharge experiments; specifically developed and calibrated DEM-CFD model and macroscopic models to address scale up issues.

Innovative aspects of the FISFine research methodology

The challenges to address require adequate methodologies, often at the frontier of research. On top of methods that can be considered standard, in the present project, we propose to make use of the following unconventional tools:

Powder characterization

-) **rain-off tests** (Zafar et al., 2015; Girimonte et al., 2018) will also be used for the determination of the cohesion of powders and powder mixtures, a procedure aimed at direct observation of the tensile strength of powders under gravity in the presence of air flow;

-) **bed collapse tests** (Girimonte and Formisani, 2014) will be employed to determine the bed defluidization and de-aeration dynamics, a process very likely relevant to container filling/discharge.

Computational modelling

-) special CFD-DEM methods for fine and polydisperse powders: **hysteretic JKR model** for Van der Waals forces and **tribocharging**/electrostatic interactions with **polarization** (Giordano et al. 2025); **drag force model for polydisperse solids** (e.g. Cello et al., 2010); *thick wall* **concept** for wall contacts of highly polydisperse solids (Alfano et al., 2024); particularly relevant for powder flow close to container and valve walls;

-) model parameters: **Bayesian calibration** of DEM material properties, e.g. for cohesion;

-) scale-up strategy: **multispecies coarse graining** for particle mixtures (Di Renzo et al., 2021), highly necessary to extend lab-scale results to pilot or industrial scales.

Macroscopic modelling

-) **Particle Segregation Model** (PSM) (Di Maio et al., 2012) for mixtures of fine or fine/intermediate size powders.

It is not expected that all the above methods will succeed in overcoming the limitations of current research in this field. Some may prove unsuccessful or inapplicable. However, using such a diverse set of unconventional tools together is a unique opportunity to provide new insights and shed light on areas that have been inaccessible until now. Therefore, there is a high likelihood of obtaining breakthrough results.

FISFine project structure

The research work is structured in four work-packages, each one dedicated strategically to one area of the project activities. As highlighted in underlined text various project activities are conceived to benefit from the highly collaborative and interactive nature of the IFPRI consortium, establishing a tight intellectual relationship with IFPRI members and exploiting the support they can provide in terms of materials, test methods and industrial experience. Detailed proposed activities, duration and expected outputs are presented in the following table.

WP1 – Powder characterization	Activity Experimental	Duration M01-M12

Task 1.1 Selection of materials and mixtures

Selection of 4 materials (with suggestions from **IFPRI** members) with the following properties:
- 1 fine powder material (model material)
- 1 fine powder material (industrially relevant)
- 1 intermediate size material (industrially relevant)
- 1 polydisperse (fine and intermediate/coarse) material (industrially relevant)

Selection of solids combinations for the mixtures and industrially relevant compositions (with suggestions from **IFPRI** members):

Sample No.	No. of components	Geldart class
1	2	A-A
2	2	C-A
3	2	C-B
4	2	C-C
5	1 (two sizes)	A-B or C-A or C-B
6	3	C-C-A or C-A-B

Task 1.2 Materials basic characterization

Determination of size, density, Geldart class, packed bed voidage, individual minimum fluidization/bubbling velocity. For each material

Material property	Principle	Instrument	Availability
Particle size distribution	Laser diffraction (ISO13320-1) or dynamic image analysis (ISO 13322-2)	Malvern Mastersizer/ Sympatec Helos/Qicpic	Yes
Particle shape	Dynamic image analysis (ISO 13322-2)	Sympatec Qicpic	Yes
Particle density	Helium pycnometry (ISO 12154) (and balance)	Quantachrome Ultrapycnometer	Yes
Fluidization properties (voidage, umf/umb)	Fluidization tests (ASTM D7743-12)	Fluidization columns	Yes

Task 1.3 Materials advanced characterization

Determination of cohesive tensile strength by rain-off tests, powder flowability indices, bed collapse tests of fine powders and powder blends.

For each material

Material property	Principle	Instrument	Availability
Aeration properties	Bed collapse (non-standard)	Bed collapse column	Yes
Cohesive strength	Rain-off tests (non-standard)	Rain-off column	Yes
Flowability	Yield loci in shear tests (e.g. ASTM D6773)	Shear cell	In partnership

Expected outputs

Selected set of materials and mixtures; Materials' basic properties; Materials' cohesion properties

WP2 – Fluidization, segregation and container filling/discharge	Activity Experimental	Duration M09-M36

Task 2.1 Bubbling fluidization tests on mixtures

Determination of packed bed voidage, fluidization interval

Task 2.2 Segregation tests on fluidized binary and ternary mixtures
  Selection of the segregation/mixing index
  Determination of the steady-state concentration profiles developing from initially mixed beds
Task 2.3 Design and build lab set-up for container filling/discharge using 3D printing
  Build a flexibly designed lab-scale container set-up (<u>with suggestions from **IFPRI** members</u>) for filling/discharge tests at different fall heights, with powder sampling for local concentration measurements.
Task 2.4 Experimental tests on filling/discharging of container with standard geometry
  Analysis of the collected data from a campaign of filling/discharge experiments of the mixtures

Expected outputs
Collection of fluidization/segregation data on binary mixtures with fines; Design and build up of lab-scale set-up for container filling/discharge experiments; data on filling/discharge experiments.

WP3 – Modeling and simulations	**Activity** Computational	**Duration** M07-M36

Task 3.1 Develop specific CFD-DEM methods and post-processing tools for the simulation of flowing and fluidized fine particles and particle mixtures across multiple scales;
  Use of open-source CFD-DEM software with advanced features specific for fine and highly polydisperse solids, including:
   - drag model for polydisperse particles   "thick wall" concept for wall contacts of highly polydisperse solids   - Van der Waals models, e.g. by JKR model with full hysteresis   - Electrostatic forces (tribocharging and electrostatic interactions, including polarization)   *Task 3.2 Calibrate model parameters based on data from characterization experiments;*
  Calibration of model parameters using advanced statistical methods (e.g. Bayesian calibration)
Task 3.3 Simulate incipient fluidization of monodisperse and binary beds of fine/intermediate-size particles;
  Determination of simulated fluidization/segregation dynamics and steady-state for different mixtures
Task 3.4 Simulate container filling/emptying processes, with focus on regions surrounding valves and openings
  Determination of the flow and segregation propensity of powder mixtures during container filling and storage.
Task 3.5 Assess the validity for fine particles of macroscopic segregation models (e.g. PSM) developed for coarse particles.
  Evaluation of the applicability of the macroscopic segregation models associated with the fluidization to mixtures involving fine powders

Expected outputs
Frontier code for physics-based modelling and simulation of fine materials and mixtures; Calibrated set of parameters for CFD-DEM simulations; simulated data of fluidization/segregation behaviour of mixtures; simulated data on container filling/discharge process; assessment of applicability of macroscopic segregation models and possible segregation prediction data.

WP4 – Synthesis and solutions	**Activity** Theoretical/experimental	**Duration** M25-M36

Task 4.1 Analysis of the collected data to gain understanding
  Collection and critical analysis (<u>after confrontation with **IFPRI** members</u>) of the combined experimental and computational results;
  Evaluation of the simulated results beyond the lab scale (heading to pilot and industrial scale)

Propose how material, process, and operation properties are linked to the likelihood and extent of fluidization-induced segregation
Task 4.2 Propose and evaluate segregation mitigation strategies
Ideation of solutions for limitation of segregation issues
Experimental tests with alternative geometries and processing options
Expected outputs
Comparative and critical analysis of experimental and computational results at lab scale; larger scale simulated results exploiting upscaled methods; propose links between material/process properties and segregation; identification of promising segregation mitigation solutions: assessment of feasibility and extent of improvement.

Critical aspects of the project activities

The research staff has long experience with multiphase processes involving granular materials. However, focusing on fines, as proposed in this project, poses important challenges in terms of successful property characterization and experimental test campaigns. The researchers are aware that fine powders can get stuck nearly anywhere, industrial fine powders can suffer from time effects (e.g. caking), they also tend to be hazardous for workers (and students!). From a simulation perspective, the situation is not easier, as fine particles lead to exponentially increasing number of particles in DEM, tiny time-steps and other issues (like particle escape from the domain, see the "thick wall" concept). Therefore, the project activities may face various hard times. To overcome them, the researchers plan to periodically seek the expert advice, comments and recommendations of the IFPRI consortium members.

Capacity of the research group

The research team includes experienced faculty professors Alberto Di Renzo, Francesco P. Di Maio, Rossella Girimonte, Daniele Sofia and Francesca O. Alfano. They gained over 20 years of experience in research on computational modelling and experimental analysis of fluidization processes, with special emphasis on mixing/segregation. One co-funded early-career researcher (e.g. PhD) will be recruited for the three-year period, plus one experienced researcher for one year.

The research will be conducted in the CheProDeS laboratory at the University of Calabria, a 220 m^2 lab hall hosting instrumentation for particle/powder characterization (PSD, density, shape, moisture, charge), sample preparation and various measurements (mills, oven, thermobalance, ion gun, Faraday cup/electrometer), equipped lab-scale facilities for fluidization tests with ambient air, hot air, ambient water, under homogeneous/bubbling, turbulent and recirculating conditions. The lab also hosts two 3D printers for AM. Computational capabilities includes a fully in-house software/hardware platform: CFD-DEM code, FEM software, CAD, MATLAB and other post-processing tools, workstations and 2 linux-based clusters (128 computing cores and 384 computing cores) for MPI-parallel execution.

In recent years, several projects shared elements with the present proposal. For example, two projects (one ongoing) and two company-funded research contracts (completed) deal with aerodispersion of pharmaceutical solid dosages for inhalation; one project (completed) and one PhD funding project (ongoing) deal with advanced green applications of fluidized bed drying; in collaborations with international colleagues and a company the group worked on electrostatics in particle technology applications. In another activity related to green energy conversion and CO_2 capture, segregation in multicomponent fluidized beds was studied by experiments and macroscopic modelling. Overall, the focus of the FISFine proposal on fine powders fits well with existing and future research trajectories.

Project timeline (three years)

The proposed Gantt chart of the activities is shown below. Activities that will be completed by the end of each year can be seen as ended at the thick lines of months 12, 24 and 36.

FISFine (IFPRI) project GANTT	Project bi-MONTHs																	
WP or Task	1-2	3-4	5-6	7-8	9-10	11-12	13-14	15-16	17-18	19-20	21-22	23-24	25-26	27-28	29-30	31-32	33-34	35-36
WP1 - Powder characterization																		
Task 1.1 Material selection	X	X																
Task 1.2 Basic characterization		X	X	X														
Task 1.3 Adv. Characterization				X	X													
WP2 - Fluidization and container tests																		
Task 2.1 Fluidization tests					X	X	X											
Task 2.2 Segregation tests						X	X	X	X									
Task 2.3 Build container set-up										X	X	X						
Task 2.4 Collection of container data												X	X	X	X	X	X	X
WP3: Modelling and simulation																		
Task 3.1 Develop model/code				X	X	X	X											
Task 3.2 Calibrazione Bayesiana							X	X	X									
Task 3.3 Simulate incipient fluidization								X	X	X	X	X						
Task 3.4 Simulate container tests													X	X	X	X	X	
Task 3.5 Assess macroscopic models				X	X	X	X											
WP4 - Synthesis and solutions																		
Task 4.1 Evaluation of complete data													X	X	X	X	X	
Task 4.2 Propose mitigation measures													X	X	X	X	X	X

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