A REGIME MAP APPROACH FOR PREDICTING THE AGGLOMERATION OF FINE WET POWDERS AFTER DRYING UNDER VARYING LEVELS OF SHEAR

IFPRI Research Project Proposal: Drying Wet Powders with Shear to Prevent Agglomerate Formation

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BACKGROUND

Wet powder drying is a unit operation used in a variety of industries, yet the many variables involved in this process make it very difficult to predict the dried powder product attributes. A particular concern for drying of fine powders is the tendency for agglomerate formation and also attrition, which are highly dependent upon the level of shear in the dryer. The extents of both agglomeration and attrition can be characterized by the change in particle size and size distribution between the initial powder and the dried powder product.

Important dried powder product attributes include particle size and size distribution, particle shape, and bulk density. Formulation properties (e.g., particle size and size distribution, particle shape, particle density, bulk and tapped density) and process variables (e.g., liquid content, temperature, shear rate, time) can all influence the product attributes.

A regime map can be a useful tool to guide formulation choices and process operation, and is an improvement upon just performing many experiments to test variables, and is a positive step toward predictive model development.[1] Regime maps for wet granulation (the process of adding a liquid binder to a powder bed, usually with some level of agitation, for particle size enlargement) were established over 20 years ago, with separate regime maps for nucleation[2] and growth.[3,4] More recently, we expanded this regime map framework for single drop granule formation in static powder beds (see **Figure 1**).[5] Two distinct mechanisms and corresponding granule shape regimes were discovered, based upon the powder bed porosity and the granular Bond number. Spreading occurred with granular Bond numbers <65,000 (roughly corresponding to surface mean particle sizes >30 μm) for all bed porosities below the minimum fluidization porosity and formed flat granules, while Tunneling occurred with granular Bond numbers >65,000 (roughly corresponding to surface mean particle sizes <30 μm) for all bed porosities and formed round granules. These two regimes in particle size, from coarser particles that naturally form a smooth, packed bed of individual particles, down to finer particles that form a fluffy powder bed comprised of dry agglomerates, are expected to be critical components in the development of a drying regime map for this proposed work.
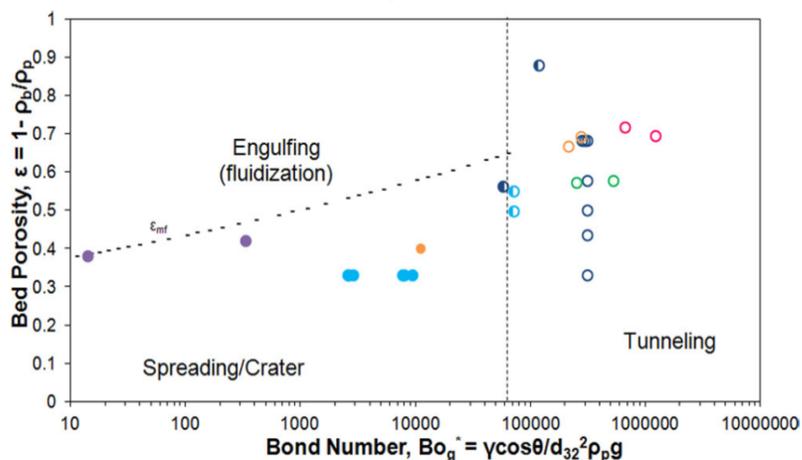

Figure 1. Granule formation mechanism regime map.[5]

PROPOSED RESEARCH PLAN

The overall goal of this research is to develop a particle drying regime map that determines the conditions under which agglomerate formation will occur in drying processes with varying shear rates. Toward this end, the following research objectives will be explored:

(**RO 1**) Raw Material Characterization and Preparation of Wet Particle Assemblies;
(**RO 2**) Tray Drying of Wet Particles [*no shear*];

(**RO 3**) Rotary Drying of Wet Particles [*low-medium shear*];
(**RO 4**) Jet Milling of Wet Particles [*high shear*];
(**RO 5**) Dried Product Characterization; and
(**RO 6**) Regime Map Development.

Details of each objective are provided in the subsequent sections, and a proposed timeline for carrying out the work for all objectives is included at the end of the proposal, in **Table 3**. More details are given for **RO 3** to demonstrate the capability of our in-house rotary drum dryer system, while the drying tests for **RO 2** and **RO 4** use standard commercial equipment.

(RO 1) Raw Material Characterization and Preparation of Wet Particle Assemblies:

The selection of materials will be done with the input of IFPRI members and will cover the two important particle size regimes (e.g., <30 μm and 30-100 μm). In addition to particle size, thorough raw material characterization is essential for elucidating the powder properties that will be relevant for regime maps. The techniques available in our powder characterization lab, outlined in **Table 1**, will be utilized for this purpose.

Table 1. Equipment and corresponding measurements used for material characterization.

Equipment	Measurement
Malvern Morphologi G3 Automated Particle Characterization System with Automated Sample Dispersion Unit	Particle size and shape, and their distributions
AccuPyc II 1340 Automatic Gas Pycnometer	Particle density
SOTAX Tap Density Tester	Bulk and tapped density

The wet particle assemblies will be prepared by mixing the powder with the desired amount of liquid and kneading until the mixture appears homogeneous. The mixtures will be prepared immediately before each test in order to minimize evaporation.

(RO 2) Tray Drying of Wet Particles [no shear]:

Oven tray drying will be performed in order to have a control drying case without shear. We have a Fisher Scientific 60L Gravity Oven in our lab that is capable of operating at temperatures up to 250°C.

(RO 3) Rotary Drying of Wet Particles [low-medium shear]:

Rotary drying will be performed in order to test the low-medium shear regimes. We will use our in-house rotary drum experimental setup that is designed for conductive and convective granular heat transfer, with particle temperature quantification via a thermal IR camera. Rotary drum systems have been used to explore mixing of fine particles, as characterized by the axial dispersion coefficient,[6,7] but little experimental work on the heating and drying of this class of materials in rotary drums is available in the literature.

Details of our experimental rotary drum setup and thermal IR camera are presented in Adepu et al.[8] The setup consists of a stainless-steel cylinder (ID = 15.24 cm, OD = 16.51 cm, L = 7.62 cm) that is closed on each end by 6 mm
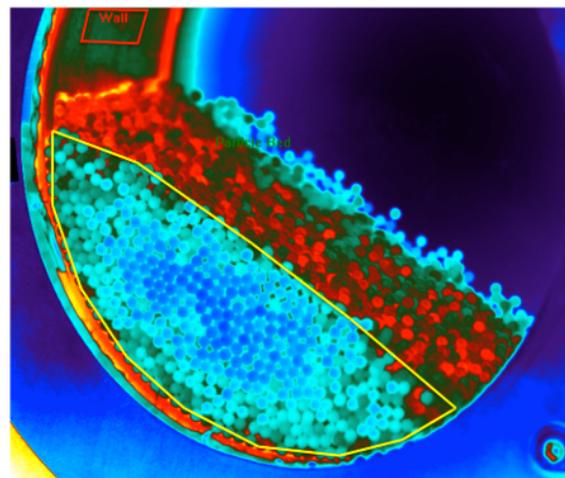
Figure 2. Sample IR camera image, with region of interest selection for the drum wall (red) and the particle bed (yellow).

thick windows. One end of the drum is closed by a sapphire window (D = 15.24 cm) that provides optical and thermal access to the interior particle bed, and the other side of the drum is closed by a quartz window (D = 16.51 cm) that provides optical access to the particle bed. Both windows are held in place using 1.27 cm thick titanium rings that are specifically chosen due to the low conductivity, thereby preventing direct contact of the drum walls with the rollers used for rotating the drum and reducing heat loss through any contact. The sapphire window is held using a 14.478 cm ID, 27.94 cm OD titanium ring, and the quartz glass is held using a 15.24 cm ID, 27.94 cm OD titanium ring. The drum rotates on the two 27.94 cm OD titanium wheels using rollers with variable rotational speed. Three heat guns are placed with equal spacing, at 120° apart, around the drum, to provide effective uniform heating. An infrared radiation (IR) camera (FLIR A6701SC) is used to capture the temperature profile through the sapphire window (see **Figure 2)**. Therefore, the sapphire window is specifically chosen to give a high transmittance to IR light, with a transmission range from 0.17 μm to 5.5 μm.

This rotary drum system was designed for conduction heat transfer via the drum walls. However, to incorporate convection heat transfer, the setup has been modified to introduce forced convection into the drum system. For the conduction setup, one side of the drum is closed using a quartz window and another side is covered using a sapphire window. For convection, besides the sapphire window that is used for IR imaging, a quartz window with holes to pump hot air into the drum is used. The quartz window used features a central inlet hole for hot air to be forced into the drum for internal heating. It also has four smaller holes around the edge to let air outside, in order to release the pressure from inside the drum. The internal heat gun is attached to an air duct that connects with pipe fittings to a temperature sensor and the air inlet port. This heat gun serves to insert a hot air stream into the drum to heat the particle bed inside the drum via forced convection. The rotary drum experimental setup, showing the placement of all four heat guns, is provided in **Figure 3**.

(a)   (b)
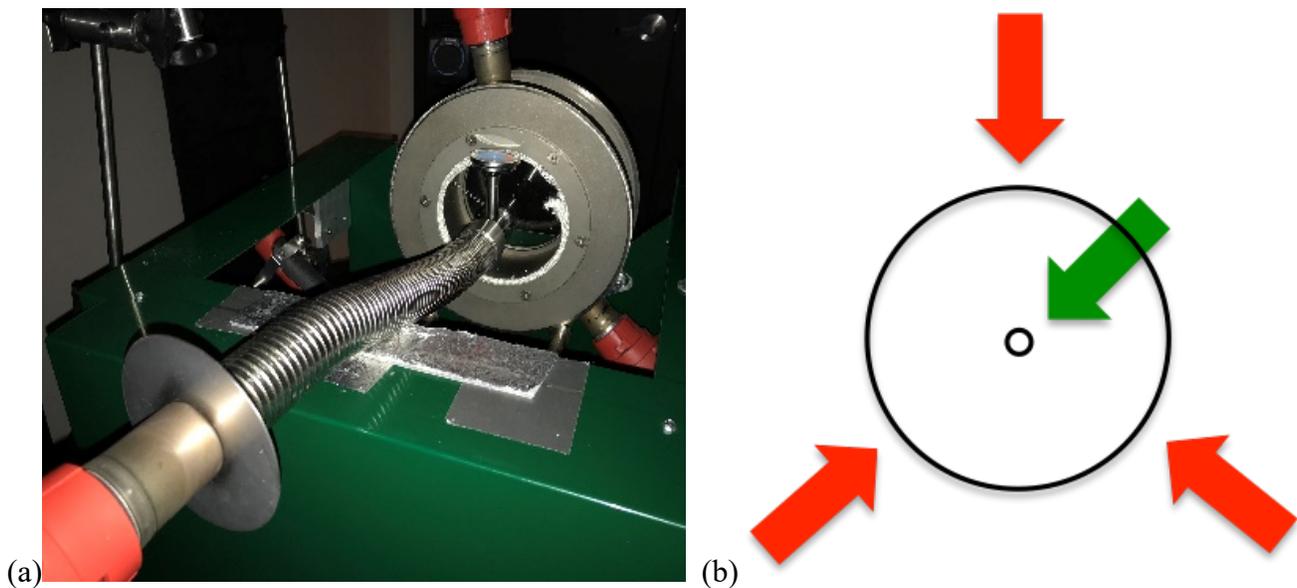
Figure 3. Rotary drum experimental setup. (a) Rear view of empty drum with air inlet duct and quartz window. The opposite end of the drum is enclosed with a sapphire window, where the thermal IR camera is positioned in order to obtain the particle temperature profile within the drum. (b) Schematic of the heat addition to the rotary drum, with the red arrows indicating external heat guns, and the green arrow indicating an internal heat gun.

(RO 4) Jet Milling of Wet Particles [high shear]:

Jet milling will be performed in order to test the high shear regime. We have a Fluid Energy Model 00 Jet-O-Mizer Mill in our lab, which can process 0.75-15 g/min of material.

(RO 5) Dried Product Characterization:

The dried powder will be characterized using the same techniques outlined in **RO 1**. A key attribute of the dried powder product is the extent of agglomeration. The product particle size, shape, and their distributions will be measured using the Malvern Morphologi G3. These product size and shape characteristics will then be compared with those of the original powder, as obtained in **RO 1**, to quantify the extent of agglomeration.

(RO 6) Regime Map Development:

The regime map will incorporate fundamental properties of the particles (e.g., particle size and shape), as well as process conditions (e.g., moisture content and shear rate), into dimensionless groups. **Table 2** provides a summary of all of the variables that will be tested, and the results will be obtained from **RO 1-5**, which will be used for derivation of the dimensionless groups and development of the regime map that will predict agglomerate formation upon drying under shear.

Table 2. Tested variables for inclusion in regime map.

	Variable
Formulation	Particle size and size distribution
	Particle shape
	Tapped and bulk density
Process	Temperature
	Shear rate
	Mass of material
	Moisture content
Product	Particle size and size distribution
	Particle shape
	Tapped and bulk density

CRITICAL UNKNOWNS

The primary uncertainties in the proposed work are: (1) the quantification of the shear rate, and (2) determination of the dimensionless groups to be used in the resulting regime map. Shear rate is variable across various equipment designs, but we strive to develop a single dimensionless group that quantifies shear rate across a variety of process equipment. For example, in the rotary drying process, there will be a balance between the shear of drum rotation versus the shear of air flow into the drum. We will not have an understanding of what this balance looks like until we actually perform the experiments. Additionally, we do not know the dominating variables in each of these drying processes before we do the experiments, so our experimental results will dictate the formation of the dimensionless groups for the regime map. Despite these unknowns, we have experience using this framework of characterizing the feed material, performing experiments looking at the effects of varying process variables, and characterizing the product material; then, we combine all of these components to determine the dominating dimensionless groups and corresponding regime map.

INTEGRATION INTO EXISTING RESEARCH PROGRAM

The proposed research combines our lab's two main research thrusts, namely single drop granulation and granular heat transfer. The regime map framework comes from the granulation project, which is currently funded by the NSF CAREER award. However, we do not currently have external funding for our granular heat transfer project, and the proposed work can leverage our existing infrastructure for this project, namely the rotary drum with the thermal IR camera.

OPPORTUNITIES FOR IFPRI MEMBER SUPPORT

The proposed research would benefit from the supply of appropriate particulate materials from IFPRI members. The two categories of particles needed are as follows: particles with surface mean sizes <30 μm, and those between 30-100 μm. Beyond these size requirements, we would be open to any type of particulate materials from relevant industries.

In addition to the supply of test materials, we would appreciate the input of IFPRI members for industrially relevant drying conditions (e.g., liquid saturation levels, temperature, drum rotation rate, etc.) to test in our setups.

PROPOSED TIMELINE

The proposed research will take place in Dr. Heather Emady's laboratory at ASU from August 1, 2022 – July 31, 2025, and the details are provided in **Table 3**.

Table 3. Timeline of research activities for the proposed work.

	Year 1	Year 2	Year 3
RO 1: Raw Material Characterization and Preparation of Wet Particle Assemblies			
Select materials of the following surface mean particle sizes: (1) <30 μm binary, and (2) 30-100 μm / Characterize materials: particle size and size distribution, particle shape, particle density, bulk and tapped density / Prepare wet particle assemblies for each drying test	▓	▓	▓
RO 2: Tray Drying of Wet Particles [*no shear*]			
Perform oven tray drying experiments and test for the effects of: (1) temperature, (2) mass of material, and (3) moisture content	▓		
RO 3: Rotary Drying of Wet Particles [*low-medium shear*]			
Perform rotary drying experiments and test for the effects of: (1) temperature, (2) mass of material, (3) moisture content, and (4) shear rate		▓	
RO 4: Jet Milling of Wet Particles [*high shear*]			
Perform jet milling experiments and test for the effects of: (1) mass feed rate, (2) moisture content, and (3) air pressure/shear rate			▓
RO 5: Dried Product Characterization			
Measure dried powder product properties, including size, shape, and their distributions, in order to quantify the extent of agglomeration		▓	▓
RO 6: Regime Map Development			
Develop regime maps that predict agglomerate formation after drying based on dimensionless groups involving the formulation properties and the process variables			▓

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