IFPRI – WELCOME & INTRODUCTION

Judith Bonsall – Unilever
IFPRI Technical Chair

IFPRI COMPETITION CAUTION

Participants shall not enter into any discussion, activity or conduct that may infringe any applicable competition law.

By way of example, participants shall not discuss, communicate or exchange any commercially sensitive information, including non-public information relating to proprietary equipment, processes and formulations.

This applies not only to discussions in formal meetings but also to informal discussions before, during and after meeting.

The recommendations coming out of this consortium are just that. Individual companies remain free to make independent, competitive decisions.

Any standards endorsed by this consortium are voluntary standards and any business practice recommendation developed are also voluntary.

MEETING ETIQUETTE

- This meeting is for Industrial Members/Industrial Guests & Consultants only – no academics or students
- Please ensure your Zoom name also includes your Company name – click on … at the top of your picture and select rename.
- We will mute all but the designated speaker – Designated speakers please unmute yourself then remember to mute yourself once your update is given.
- If you have a question please raise your hand via the zoom button, or put it in the chat section – Willie, Jim & I will be monitoring this during the meeting. Click on Participants Panel then click raise hand at the lower RHS
- We will try to keep to the time allowed, but forgive us if we are not quite as good as usual!

	Sunday	Monday	Tuesday	Wednesday	Thursday
am	Members only business meeting	Posters from current Technical Program	Review Presentations & Round Robin/Workshop Update	Refining Ideas for future Technical Program Briefs Scientific Screening and pre-selection of Proposals for future Technical Program	Rank order vote for next years new Technical Program. Selection of Project Briefs, Review Briefs & Workshops to progress
pm	Presentation From Current Technical Program	Posters From Current Technical Program & Feedback	Ideas session to generate new Project Briefs for Future Technical Program	Non- IFPRI funded Technical Presentations & Academic Poster Session	Members only business meeting Final Budget Decision Making

IFPRI MEETINGS – THE NORMAL PROCESS FOR TECHNICAL MEETING

Current Program Activities

Scene setting presentations

In-depth poster talks

Review presentations – chapter outlines

Feed back and critique of the projects & Reviews

Updates from consultants

Future Program Activities

Ideas generation – projects/reviews/workshops

Idea refinement and brief generation

Voting for briefs (n+1)

Discussion on merits of new project proposals

Voting for new proposals

PROJECT PHASING- 2019/2020 17 PAID PROJECTS, 1 COLLABORATION GRANT PROJECT

	Project	Research Associate	Institution	End	Term
1	Creating Tuneable Agglomerates via 3D Printing	K. Hapgood	Monash University	2020	2
2	Prediction of Segregation	J. McCarthy	U. Pittsburgh	2020	2
3	Flowability Assessment of Weakly Consolidated Particles	C. Hare A. Hassanpour	U. Surrey	2020	2
4	Molecular Self-Assembly	U. Wiesner	Cornell U.	2022	2
5	Dry Powder Rheology	K. Daniels	NCSU	2022	2
6	Model-Based Control of Crystallisation	Z. Nagy	Purdue U.	2023	2
7	Crystal Shape Prediction	M. Doherty	California	2021	1
8	Powder Mixing Rules	I. Govender	Kwazulu-Natal	2021	1
9	Cake Filtration	U. Peuker	Freiberg	2021	1
10	Wetting and Dispersion of powders	Gaiani	Lorraine	2022	1
11	Slurry & Paste Rheology	E. Koos	KIT (Karlsruhe)	2022	1
12	Powder Adhesion to Metal Surfaces	C. Sinka	Leicester Uni	2022	1
13	Characterisation of spray nozzles	N. Ashgriz	Toronto	2022	1
14	Model-based Design of Granular Products	R Smith	Sheffield	2023	1
15	Precision powder feeding: Theoretical understanding and predictive model to link material properties to performance of twin screw feeders	P. Nott	Indian Institute of Science	2023	1
16	Bridging the gap between model and industrial colloidal formulations	J. Vermant	ETHZ	2023	1
17	A Systems Engineering Approach to Dry-Milling with Grinding Aid Additives	A. Kwade	TU Braunschweig	2023	1
18	SIF collaboration	L. Hsiao	NC State	2021	1 yr

IFPRI
International Fine Particle Research Institute

Model Assisted Design of Granular Products

Linking Process and Product Models for Wet Granulation

Rachel Smith
Peyman Mostafei

The University Of Sheffield. Chemical and Biological Engineering

Help Transform Tomorrow.

Christophe Grosjean (Syngenta)

Modeling of screw feeder performance

Prabhu R Nott

Aashish Kumar Gupta

Indian Institute of Science
Bangalore

IFPRI funded project commenced on 21 October 2019

Poom Bunchatheeravate (Vertex)

ETH zürich

Simplified industrial dispersions

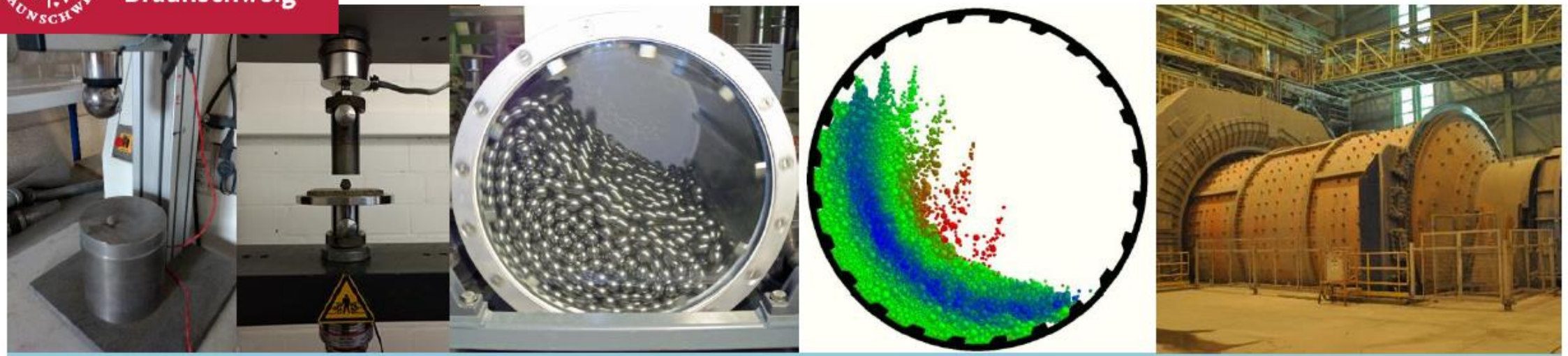
Luico Isa and Jan Vermant

Department of Materials. ETH Zürich - ETH Zürich, CH

Scott Brown (Chemours)

Technische Universität Braunschweig

iPAT
Institut für Partikeltechnik



IFPRI

A Systems Engineering Approach to Dry-Milling with Grinding Aid Additives

Anderson Chagas, Sandra Breitung-Faes, Arno Kwade

Jarrod Hart (Imerys)

A MULTISCALE STUDY OF POWDER RECONSTITUTION

Claire GAIANI & Jeremy PETIT

Tristan FOURNAISE

Pr. Claire GAIANI - Université de Lorraine
LIBio – Laboratoire d'Ingénierie des Biomolécules
2 avenue de la Forêt de Haye - BP 20163
54505 Vandœuvre-lès-Nancy - FRANCE
Tél. : +33(0)3 72 74 41 11 - Fax : +33(0)3 83 59 57 72
claire.gaiani@univ-lorraine.fr
http://libio.univ-lorraine.fr/

Vincent Meunier (Nestle)

KU LEUVEN

Dynamic and structural investigation of capillary suspensions

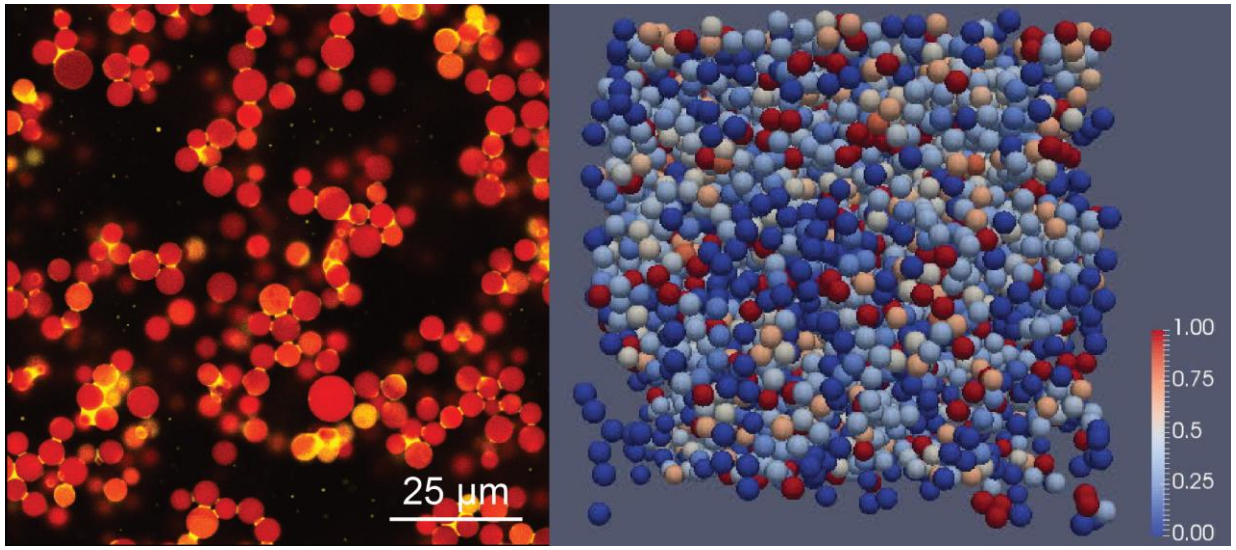
IFPRI Annual Meeting 2020

Jens Allard and Erin Koos

KU Leuven
Department of Chemical Engineering
Section Soft Matter, Rheology and Technology

Jan Wieringa (Unilever)

Erin Koos, Jens Allard:
Dynamic and thermodynamic structural investigation of capillary suspensions



Liaison feedback on IFPRI AGM 2020

Jan Wieringa (Unilever),

Guylaine St. Jean (Energizer)

Simon Greener (P&G)

Findings

Progress - relation between structure and rheological properties

- Article: amount of 2nd liquid and structure –> z, c -> G' and G''
- Experiments: step change in particle loading and shearing
  - Different behaviors -> new questions to be analysed
- Issue: 2nd liquid spread differently than expected, did not form proper bridges.
  - Attributed to surface roughness
    -> influenced hydrophobicity of the particles.
    -> future work on particle roughness
- Improved image analysis tools for rough particles and 2nd liquid spread in a ring shape.
- Made a start to work with rods instead of spheres

Conclusions:

- Good progress
- Propose to combine results in a comprehensive overview or regime map

Characterization of Spray Drying Nozzles at Industrially Relevant Conditions

Jerry (Siyu) Chen

Nasser Ashgriz

Isaac Jackiw

ARR-02-15

May 25, 2020

UNIVERSITY OF TORONTO | Department of Mechanical and Industrial Engineering

IFPRI International Fine Particle Research Institute

Joe Bullard (Vertex)

IFPRI Question / Comment	Edited Response from Nasser Ashgriz
Application of models to rotary atomizers?	Models are specific to pressure swirl and 2-fluid nozzles. Centrifugal forces, etc., are not included.
What are the limits of correlations in terms of viscosity / other rheological properties?	Current work is most developed for fluids up to ~100 cP (n.b.: Some of the work presented includes fluids of up to ~350 cP.) Higher-viscosity systems are in scope and work will begin on them in coming months.
How can PFD be implemented on a commercial system?	(Reply assumes questioner meant "PDF" and lists following applications: • Offers information on process yield, indicating amount of small/large particles • A quality check on proper functioning of a given nozzle. (i.e., knowing the PDF of a new/well-behaved nozzle can serve as a control for scratching, wear, etc. that could change nozzle behavior.) • Input to CFD models • Indicator of effect of fluid properties on atomization, for example if an increase in viscosity results in a bimodal distribution of large/small particles but an unchanged average size.
Have tests with the high pressure nozzle included air flow in the chamber?	No. This is out of scope of the current work but is something that could be added in the future. Current chamber does not have this capability.
How many replicates per condition?	**Pressure swirl:** 2 replicates for Whirljet nozzles with water, 500 images taken at each position to generate SMD value. Dv10, Dv90, D32 monitored until they fluctuate less than 2%. **Two-fluid**: 5 repeatability tests performed for a specific set of conditions to assess measurement variability. Each test on Malvern conducted for 1-2 min, sampling every second, with average values reported. Standard deviation is generally negligible.
How might air flow changes impact the droplet formation? (straight down vs. swirling air (same direction as nozzle swirl or opposite direction of nozzle swirl)	Atomization is completed in the two-fluid nozzle by about 3mm downstream of the nozzle. For swirl nozzles, using low viscosity fluids, the atomization is completed with 5mm. However, increases in solution viscosity may extend the liquid sheet to 12mm, which breaks to generate ligaments. It may take more than 10cm for the ligaments to breakup and form droplets. This is a long distance, during which the external air flow can have a significant effect on the final droplet size distribution. In the ligament phase, and during the secondary breakup processes, the type of air flow (e.g., swirling, downward, etc.) can change the ligament breakup, and subsequently, change the final droplet size distribution.

IFPRI Question / Comment	Edited Response from Nasser Ashgriz
Any insights into what might happen if the nozzle were to spray upwards instead of down?	• **Pressure Swirl**: The velocity at the nozzle exit is of the order ~10 m/s. The orifice diameters of the nozzles are of the order 1 mm. The Froude number (Fr=u/√Lg), which represents the ratio of the inertia to the buoyancy, is of the order of 102 for the swirl nozzle. Therefore, the inertial force is the dominating effect close to the nozzle, and the effect of gravity on the atomization is negligible. • **Two-Fluid**: Lowest value of Froude number is of order 103 (1750), therefore, again, the gravity effects on the atomization process are negligible. Atomization by the high speed gaseous flow is the dominant effect. • **Far from the nozzle**, and in the case of upward moving spray, the larger droplets may begin to fall back towards the nozzle. This may change the surrounding conditions for the atomization, and also spray flow by altering collision, breakup and coalescent events, which may change the droplet size distribution.
Does the composite image show the droplets in the centre of the hollow cone and those in the "Spray Region" at the cone walls?	The image shows the snapshot taken from the surface of the spray cone, and not the cross sectional plane.
When plotting a size distribution is it possible to account for around ~90% of the spray mass being in the outer cone?	Most of the mass is on the outer cone, however, the droplets quickly disperse and move in all directions. In particular, in a closed chamber like ours, the flow circulation plays a large role in the droplet dispersion. In our cases, we see a very large mass flow even at the centre of the spray, due to secondary air dispersion. Therefore, in general, even though one can say that the initial mass flow is mainly at the cone, this cannot be extrapolated to the mass distribution further downstream.

Self-Assembled Monolayers as Nucleating Surfaces to Study Early Formation Pathways of Crystal Polymorphs

Hui Du, Lara A. Estroff and Uli Wiesner

Materials Science and Engineering

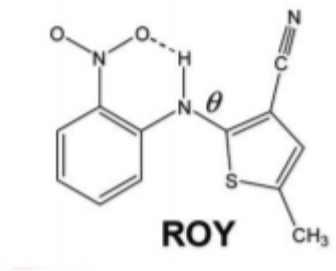
Hui Du

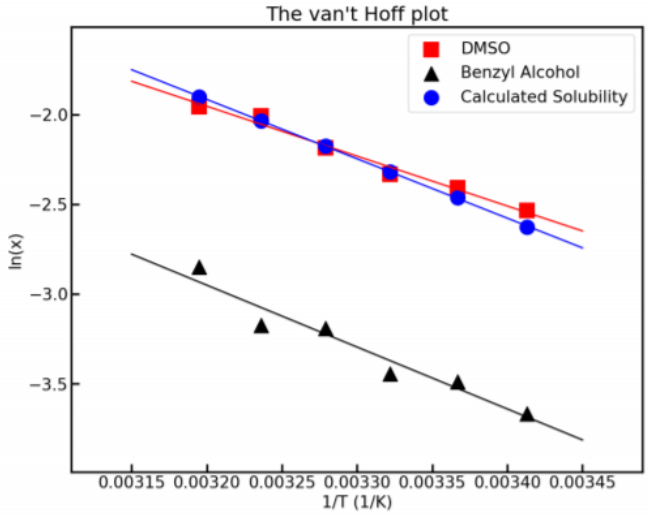
ubw1@cornell.edu

IFPRI 2020 AGM

John Hone (Syngenta)

Switch to ROY (9 forms) and Supersaturation Control

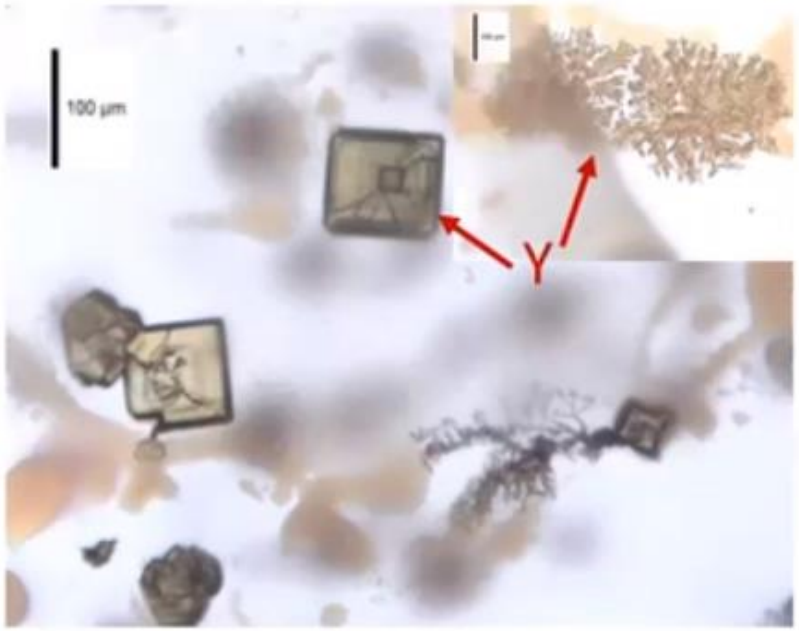




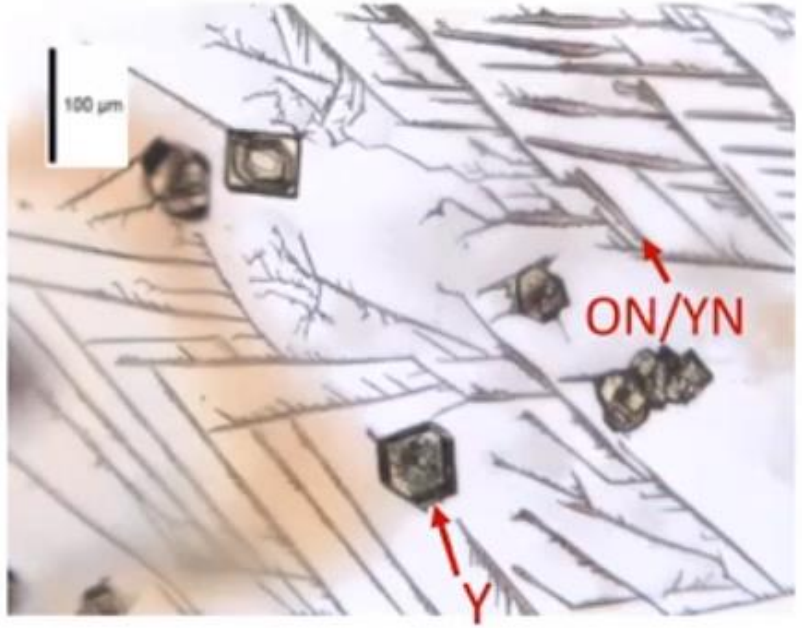
Solubility measured for supersaturation knowledge & control

CCDC: QAXMEHxx

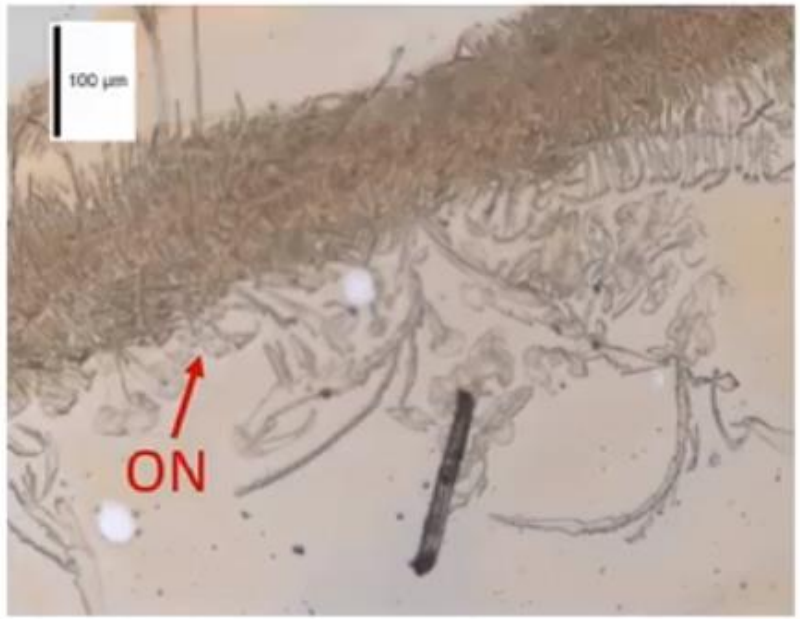
Multiple forms seen on glass slides from benzyl alcohol



S=1.5



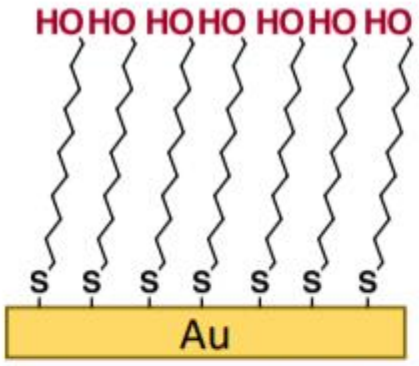
S=2



S=2.5

SAMs

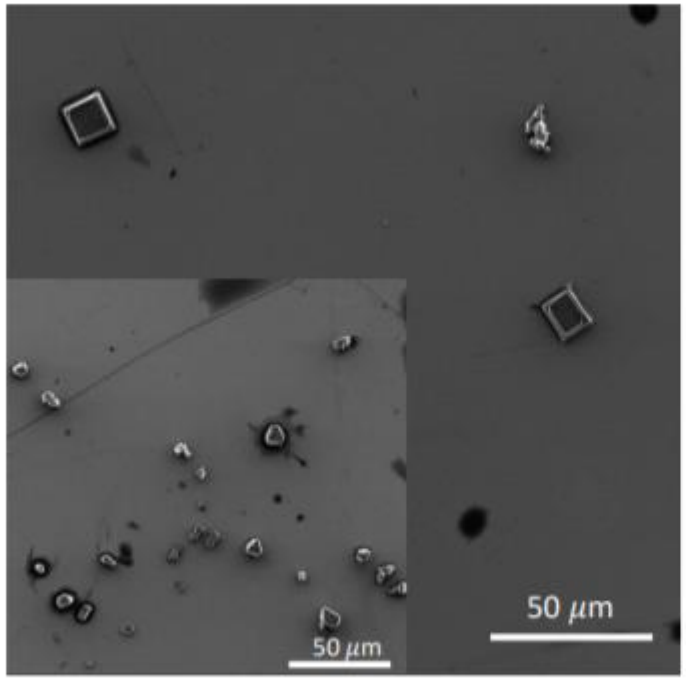
No crystals seen on –CH_3 SAM in benzyl alcohol

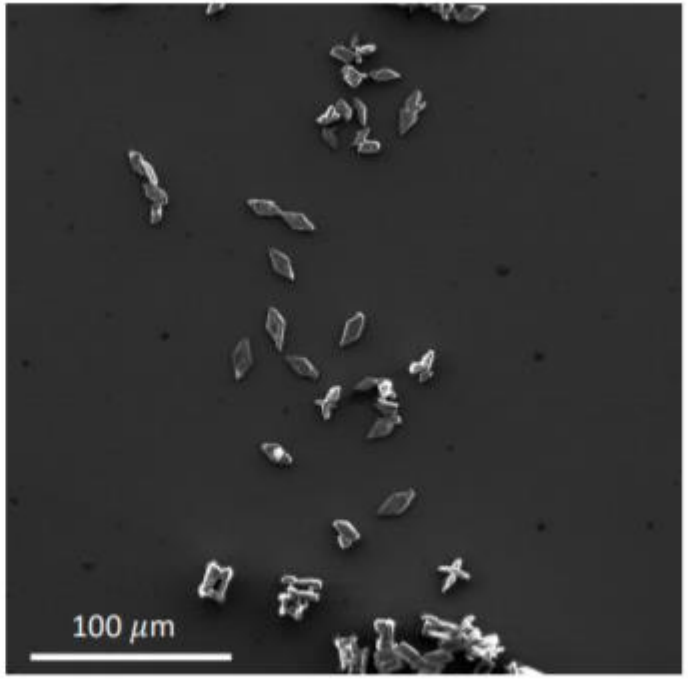


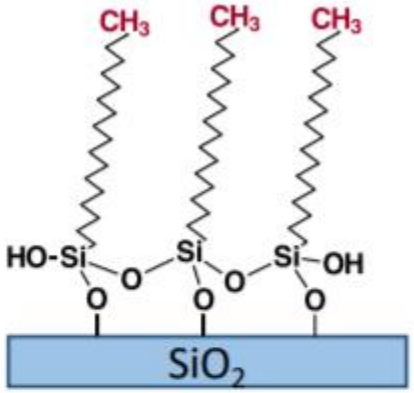
Solvent: Benzyl Alcohol

(2) **Y** $P2_1/c$
mp 109.8 °C
θ = 104.7°

(9) **YT04** $P2_1/c$
mp 106.9°C
θ = 112.8°







Solvent: Toluene

(1) **R** P-1
mp 106.2 °C
θ = 21.7°

Comment 1:

Would be interested in seeing more surface characterization SAM (i.e maybe contact angle or hydrophobicity, or AFM for surface feature)

At the very beginning of this IFPRI project, we checked whether our SAMs are high quality by reproducing earlier results of how specific SAMs lead to nucleation and growth of inorganic crystals with particular orientations. In particular, we showed that calcite ($CaCO_3$) crystallized on –COOH terminated SAMs is oriented along the (012) facet, exactly how is has been described in the literature (JACS 129 (2007), 5480). But we can certainly generate some additional AFM images to further demonstrate our SAM quality.

Comment 2:
How easy is it to put different functional group on SAM, that way we can have access to more energy level. I think in the video there were only 4 different Sam, how easy/difficult to make other types

We definitively want to study more different SAMs. As pointed out in the video, the results we got were first proof-of-principle results that we got in just before the Covid-19 pandemic shut all labs down at Cornell. Now that we are reopened again (since last week, at 1/3 of the usual occupancy only, however), we will continue to look at other SAMs and their effects on the polymorph selectivity of ROY. Their preparation is straight forward, so not a major time-limiting step.

I think it would be great to start to link the data to the chemistry. Do you have any plans later to do that, perhaps with MD simulations or similar?

The current continuation plan is to basically get the details straightened out, as discussed/described in the talk. We need to do the x-ray in order to assign the polymorphs and will also do Raman on the samples. Once we have a couple of clear "winners", **my thought was to then try to go the synchrotron again in order to look at the early formation stages, which is what the IPFRI brief really was charging us with.** Do you think this is the right way to think about it?

Mike Doherty's work has clear links here (not so much the IFPRI project) but the work on understanding morphology – if he's willing you could understand which faces are attached to the surface.

Concerning Mike Doherty: **It would indeed be nice** to get him to look at this. But for one, maybe that should wait until we have some synchrotron data on early formation stages? Also, **I am not sure whether I can talk him into this**, as someone needs to pay for the student on his end seriously looking into this. In academia, that is always a bit of a problem…

Controlling Rheology via Boundary Conditions in Dense Granular Flows

Farnaz Fazelpour, Karen E. Daniels
North Carolina State University

IFPRI
International Fine Particle Research Institute

NSF

NC STATE UNIVERSITY

Marty Murtagh (Corning/Cornell)

Controlling Rheology via Boundary Conditions in Dense Granular Flows; Karen Daniels, et al.

Updates:

- Cambridge: Nathalie is no longer funded, so her work was not include in my report. But conversations, if not direct collaboration, are ongoing
- If the video did not seem "new" it was because she talked about the results existing in preliminary forms last year, while continuing to work on them to solidify and quantify them. Since there were only 2 months from the December report when the group left the lab, she did not get a chance to complete the work we had intended to complete this spring

Controlling Rheology via Boundary Conditions in Dense Granular Flows; Karen Daniels, et al.

Question: I cannot recall why her apparatus cannot rotate faster

- **Answer:** The motor + gearbox cannot drive our flow any faster than we have shown. This is why we involved Nathalie Vriend in the collaboration, and successfully took faster data in her apparatus. Our planned Year 3 (to start this fall) was scheduled to be the construction of a hopper flow that allows for intermediate flows here at home. Some of the work this past year has been development-work on the imaging system that will allow us to collect higher-speed data. This will need a different lighting and camera setup than existed in the previous apparatus. I have solved the expense of a high-speed camera by borrowing it from another project in the group. So, faster speeds are coming but require us to be back in the lab in order to achieve.

Controlling Rheology via Boundary Conditions in Dense Granular Flows; Karen Daniels, et al.

Question: what are the limits of the stiffness of the outer wall of Karen's apparatus? Can it be released in order to provide a free (or almost free) surface?

- **Answer:** Stiffness: it is adjustable-stiffness in principle (but we've only made real experiments with 1 stiffness), but then for the roughness experiments we had to move away from using that boundary, since it doesn't allow for roughness to be controlled independent of stiffness. No: it cannot be "released" as it is composed of leaf-springs, each of fixed stiffness.

Controlling Rheology via Boundary Conditions in Dense Granular Flows; Karen Daniels, et al.

Question: I would appreciate some discussion about the impact of the missing data, and what might be done to fill in those gaps.

- **Answer:** There is a lower limit to *any* force technique, and the use of photoelasticity for force measurements will not do worse than the spring boundaries. The real reason for using it its that it has the significant benefit (once we get everything calibrated) of measuring the full stress tensor as a function of radial position, rather than us just a fit of two endpoints a presumed stress function, as was done in Zhu Tang's work. This fills in a gaping hole in the spatial data (we don't have a serious gap in force resolution that causes us particular problems).
- **Answer:** Using this technique the same types of measurements we did with Nathalie Vriend become possible: that work was based on her fast camera + apparatus, and our photoelastic analysis code. Once we go to to more collisional flows, the low-force resolution could become a problem, as it was near the surface of Nathalie Vriend's flows, but we don't expect this a problem for us since we won't be studying a free surface flow as she was. This is another reason for the choice of hopper-like geometry for our upcoming experiment.

Controlling Rheology via Boundary Conditions in Dense Granular Flows; Karen Daniels, et al.

Question: There was a brief mention of collaboration with Karen Hapgood (3-d printed coffee beans?) but no details were provided.

- **Answer:** Yes, there is an ongoing collaboration with Karen Hapgood, in which we are working together to interpret the (complicated) photoelastic signals she and her group obtain from 3D printed photoelastic grains. The conclusion so far: this can be done, but there are some pitfalls that Farnaz and I have been working to help them understand. It may be necessary for them to go back into the lab to finalize some pieces. As for why I didn't give more details: since the lead authors on the study are from Karen Hapgood's team, I figured she would report on this rather than us. I'm not sure whether this is IFPRI-funded on her end, however?

Controlling Rheology via Boundary Conditions in Dense Granular Flows; Karen Daniels, et al.

Question: What is working well?

- **Answer:** The main apparatus runs just fine in its old configuration, and short lab trips suffice to go in to start/stop runs that are similar to existing data
  - Caveat: changing to different wall to switch roughness is more time-consuming and needs to be more carefully planned with the rules (below)
- **Answer:** We have received permission to conduct duration-limited and occupancy-limited research in our lab rooms, wearing masks, as long as lab safety hazards aren't a problem (they aren't)
- **Answer:** Since the main apparatus is in its own small room, occupancy = 1, but it's ours to allocate.
- **Answer:** Current plan: go in ~ once a week, collect a new run, analyze it, repeat

Controlling Rheology via Boundary Conditions in Dense Granular Flows; Karen Daniels, et al.

Question: *What are Potential Roadblocks?*

- **Answer:** Data collection and analysis and discussion are not quite proceeding at a normal rate, including because turn-around time on glitches is lengthened
- **Answer:** The main apparatus in its old configuration does not have good enough lighting to collect high-quality data: we will need to upgrade the lighting to provide separate red (unpolarized) and green (polarized) channels.
  - The red/green LED order has now been placed, and building the new lighting setup should be possible within the constraints above.
  - We're uncertain how to implement the polarization until we try a few things in the lab.
  - Hopefully, we don't need to use the machine shop to complete construction (not currently operating).
- **Answer:** The number of COVID-19 cases in NC is currently heading back towards ICU crisis levels, so it remains possible the our access to the lab will once again be curtailed. Getting some data onto disk now is our best protection against that possibility.

A Holistic Approach for the Model-based Control of Crystal Size, Shape and Purity in Integrated Batch and Continuous Crystallization - Wet Milling Systems

Botond Szilagyi, Zoltan K. Nagy

School of Chemical Engineering
Purdue University, West Lafayette, IN

PURDUE
UNIVERSITY

Paul Mort (Purdue)

Investigating the Effect of Solvents and Impurities on Crystal Growth

IFPRI AGM 2020

Michael F. Doherty and Tobias Mazal

CHEMICAL ENGINEERING
UC SANTA BARBARA

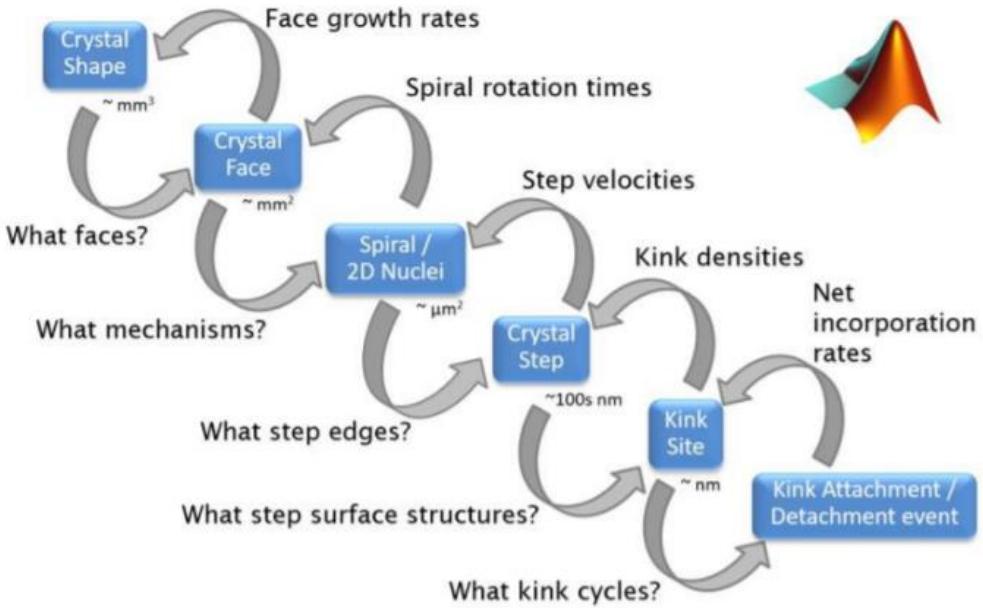
John Hone (Syngenta)

Before getting technical…

Is the model (ADDICT) available for industrial members? If so – how?

ANSWER: We are currently debugging the latest version, ADDICT 3.11. I am hoping that we can distribute it later this year.

I have a proposal for IFPRI members to consider. Once we have finished developing a really robust version of ADDICT 3.11 we can make a distribution and **follow up with a live webinar on getting started and using ADDICT as part of your work flow.**

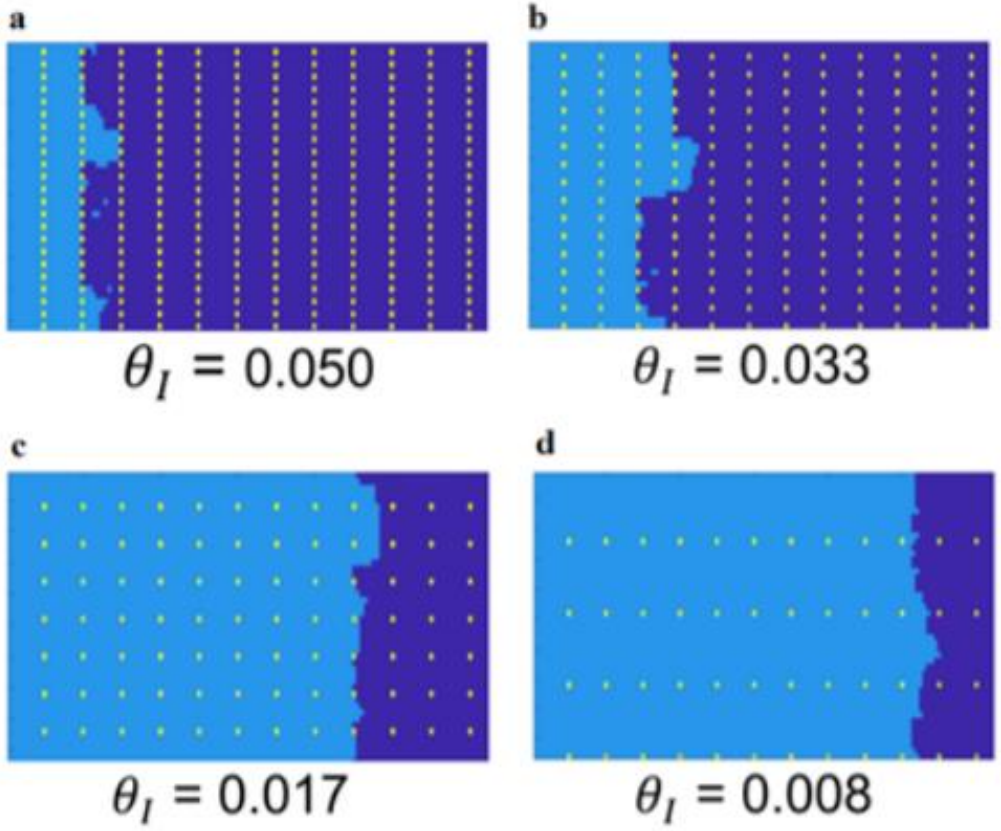


Testing Step Pinning Theory of Cabrera & Vermilyea with Kinetic Monte Carlo Simulations

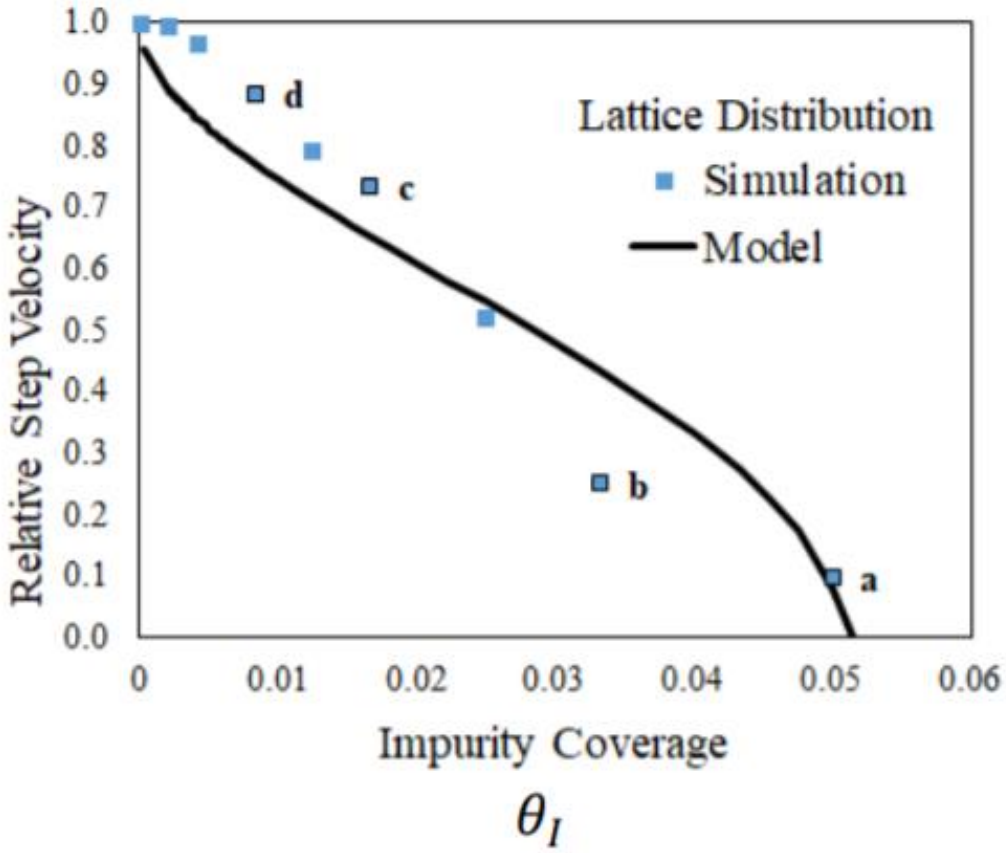
$$v = v_\infty (1 - 2 l_c d^{1/2})^{1/2}$$

Critical Length of Step

Density of Adsorbed Impurities



Good agreement with regular impurity spacing



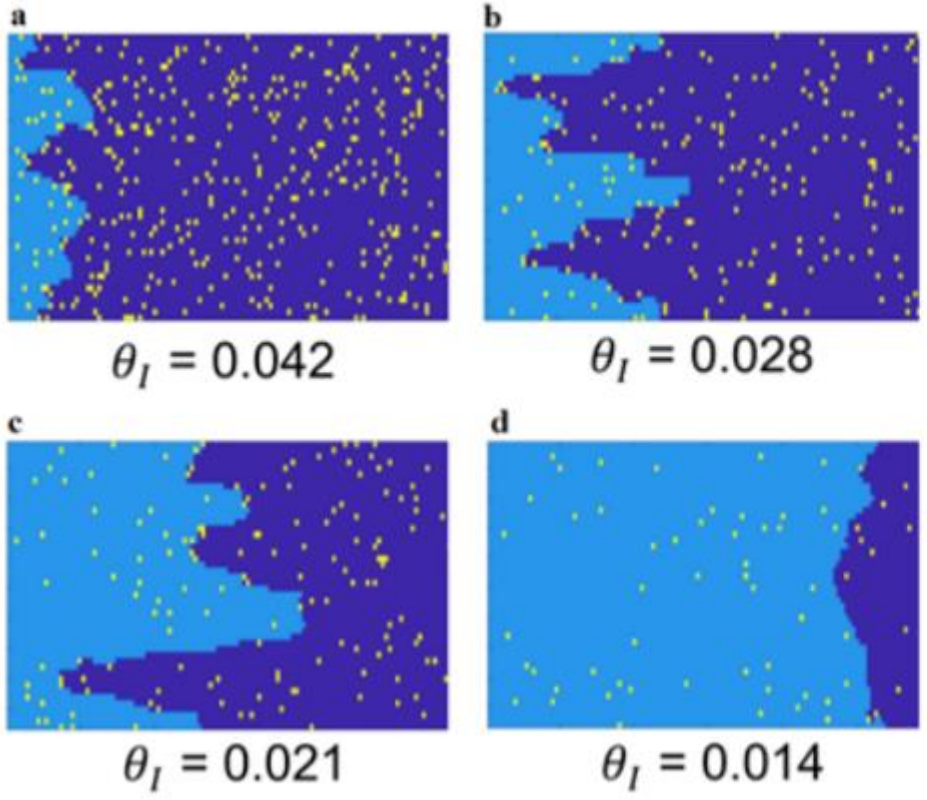
Testing Step Pinning Theory of Cabrera & Vermilyea with Kinetic Monte Carlo Simulations

$$v = v_\infty (1 - 2 l_c d^{1/2})^{1/2}$$

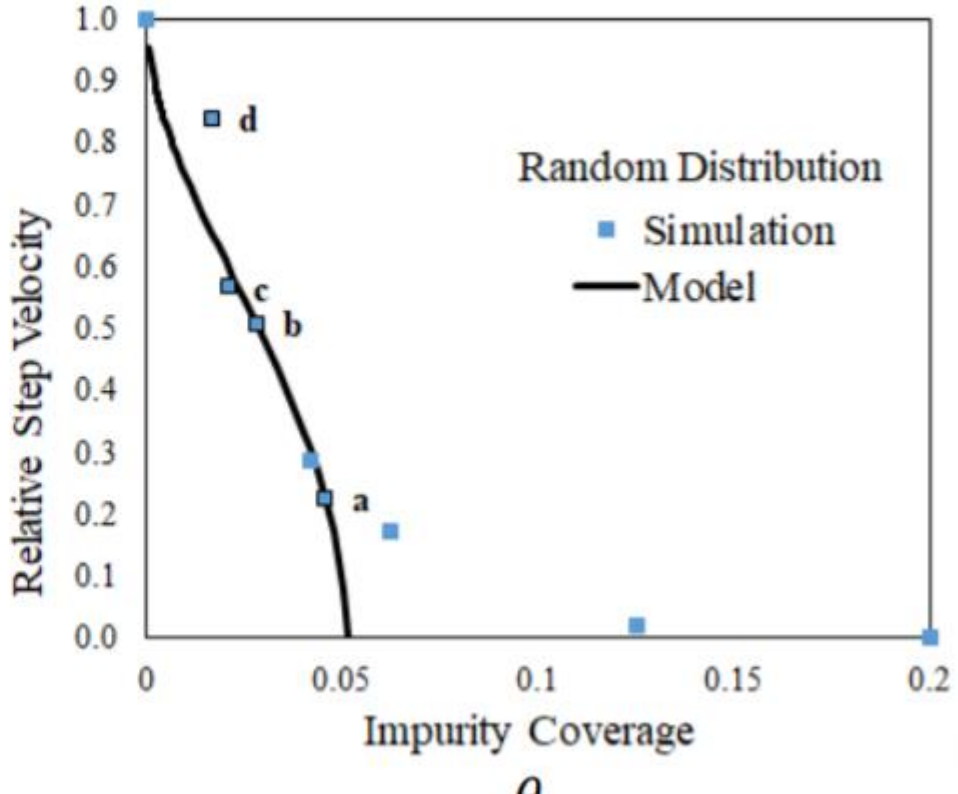
Critical Length of Step

Density of Adsorbed Impurities

$$d \propto \theta_I$$



Discrepancy with random impurity spacing at high impurity levels on terrace – checking the error



1. How does the model determine the bond energy between the surface and molecule? Is it calculated specifically for each system and face or different bond energies are used to create a map?

ANSWER: **We calculate the kink, edge, and terrace energies (between growth unit (GU) and crystal surface) directly from the solid-state force field model** (e.g., Amber-GAFF). They are calculated separately for every GU at every kink site, edge site and terrace site at every edge, on every face.

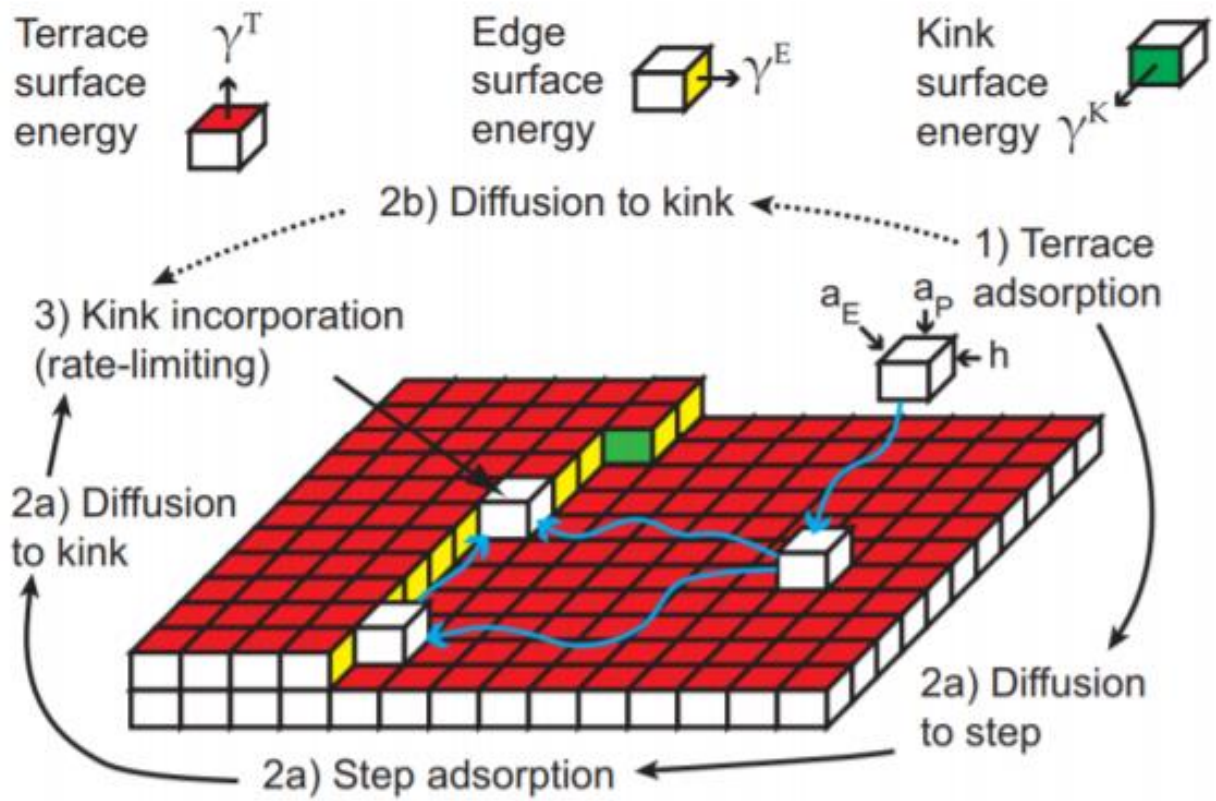


Image adapted from Tilbury et al. (**2016**)

How are the ΔU and ΔW calculated (slide 10, attachment/detachment rate expressions)?

ANSWER: **ΔW is calculated as in the previous answer**, from the force field. ΔU is primarily determined by the nature of the solvent (desolvation energy) and is believed to be approximately the same at every site. **Therefore, it cancels from the relative growth rates** – which are the ones that matter for morphology modeling.

	Attachment Rates (1/s)	Detachment Rates (1/s)
Edge ↔ Kink	$j^{+}_{K,i} = k^{+}_{K,i}\ \theta_{E,i} = v_0 \exp\left(\frac{-\Delta U^{\ddagger}_{K,i}}{k_B T}\right)\theta_{E,i}$	$j^{-}_{K,i} = k^{-}_{K,i} = v_0 \exp\left(\frac{-(\Delta U^{\ddagger}_{K,i} + \Delta W_{K,i})}{k_B T}\right)$
Terrace ↔ Edge	$j^{+}_{E,i} = k^{+}_{E,i}\ \theta_{T,i} = v_0 \exp\left(\frac{-\Delta U^{\ddagger}_{E,i}}{k_B T}\right)\theta_{T,i}$	$j^{-}_{E,i} = k^{-}_{E,i}\ \theta_{E,i} = v_0 \exp\left(\frac{-(\Delta U^{\ddagger}_{E,i} + \Delta W_{E,i})}{k_B T}\right)\theta_{E,i}$
Terrace ↔ Kink	$j^{+}_{KT,i} = k^{+}_{KT,i}\ \theta_{T,i} = v_0 \exp\left(\frac{-(\Delta U^{\ddagger}_{K,i} + \Delta U^{\ddagger}_{E,i})}{k_B T}\right)\theta_{T,i}$	$j^{-}_{KT,i} = k^{-}_{KT,i} = k^{+}_{KT,i} \exp\left(\frac{-(\Delta W_{E,i} + \Delta W_{K,i})}{k_B T}\right)$
Solution ↔ Terrace	$j^{+}_{T} = k^{+}_{T}\ x = v_0 \exp\left(\frac{-\Delta U^{\ddagger}_{T}}{k_B T}\right)x$	$j^{-}_{T,i} = k^{-}_{T}\ \theta_{T,i} = v_0 \exp\left(\frac{-(\Delta U^{\ddagger}_{T} + \Delta W_{T})}{k_B T}\right)\theta_{T,i}$

Is Addict using forcefield, DFT or COSMOtherm model?

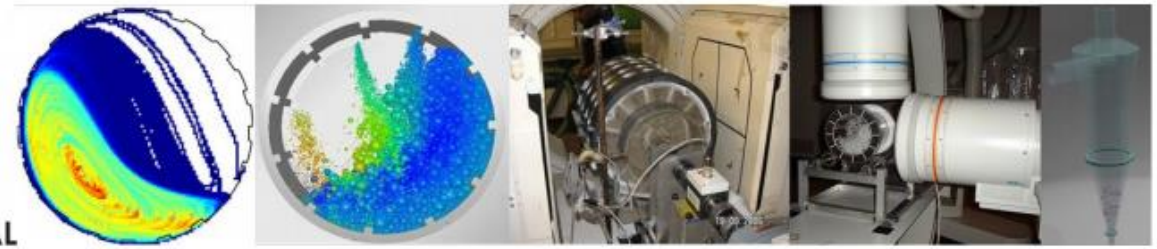
ANSWER: **Force field**. For solution-grown crystals the kink energies need to be modified to account for solvent interactions, and this is where COSMO or another solution-solid interface model is needed.

Does the model consider only the growth of a step on a face? How are new steps created?

ANSWER: **Yes, the growth models are driven by the growth rate of steps**, appropriately embedded into a spiral growth or 2D nucleation & growth mechanism. The steps are created by either screw dislocations (spiral growth) or 2D surface nuclei.

How does the Kinetic Monte Carlo model incorporate the critical length of a step (l_c).

ANSWER: **The KMC model has absolutely no knowledge of critical length** – it is not part of the simulation method in any way. KMC only knows about attachment and detachment of growth units at surface sites. This is also true of experiments – an experiment only knows about attachment and detachment of growth units at surface sites. Only the mechanistic models, such as Cabrera & Vermilyea, use such concepts as critical length. We are using KMC simulation to test the mechanistic models for the very reason that it does not know about critical length, etc. You can think of KMC simulations as a “highly controlled experiment.”



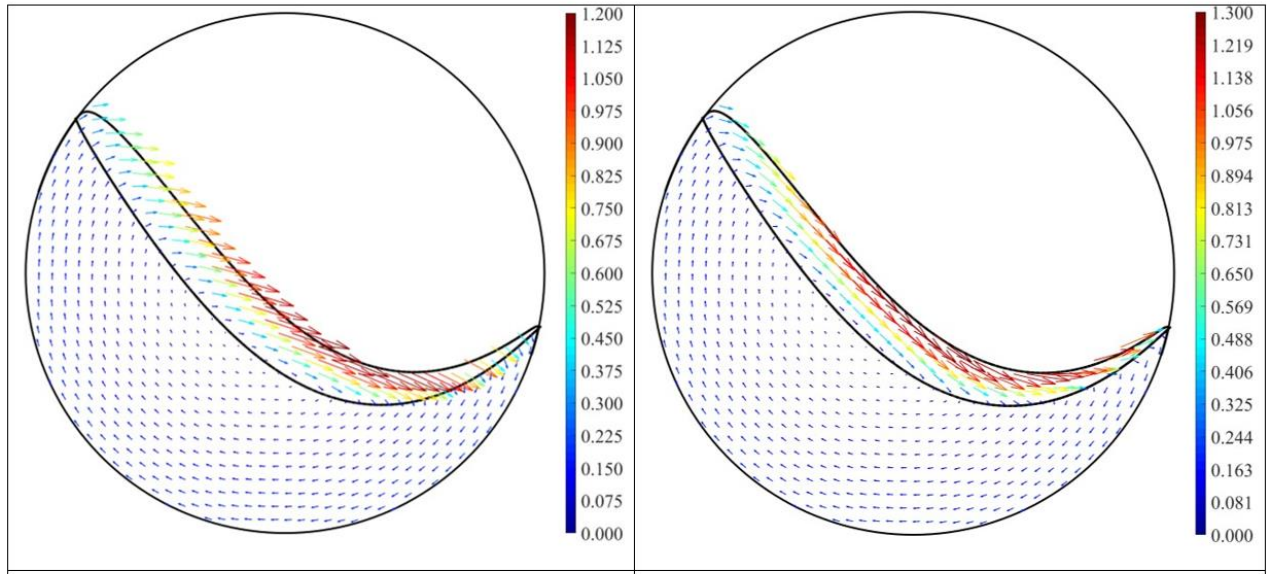
SCALE-UP RULES FOR MIXING MECHANISMS IN ROTATING DRUM FLOWS

By

Indresan Govender & Taswald Moodley

International Fine Particle Research Institute

Poom Bunchatheeravate (Vertex)/Massih Pasha (Chemours)



Hung et al (2016)	Proposed model

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Detailed insight into microscopic phenomena using 3D-tomography data to develop a better model for dead-end filtration

E. Löwer, M. Brockmann, Dr. Th. Leißner, Prof. Dr.-Ing. Urs A. Peuker

Guenter Stroh (Evonik)

IFPRI Project Abstract

Detailed insight into microscopic phenomena using 3D-Tomography data to develop a better model for dead-end filtration

Principle Investigator(s): Thomas Leißner, Urs A. Peuker
PhD-Student(s): Mashia Brockmann, Erik Löwer
Affiliation(s): TU Bergakademie Freiberg

Project Start Date: 01.10.2017
Abstract Date: 29.05.2020

Project Objective:

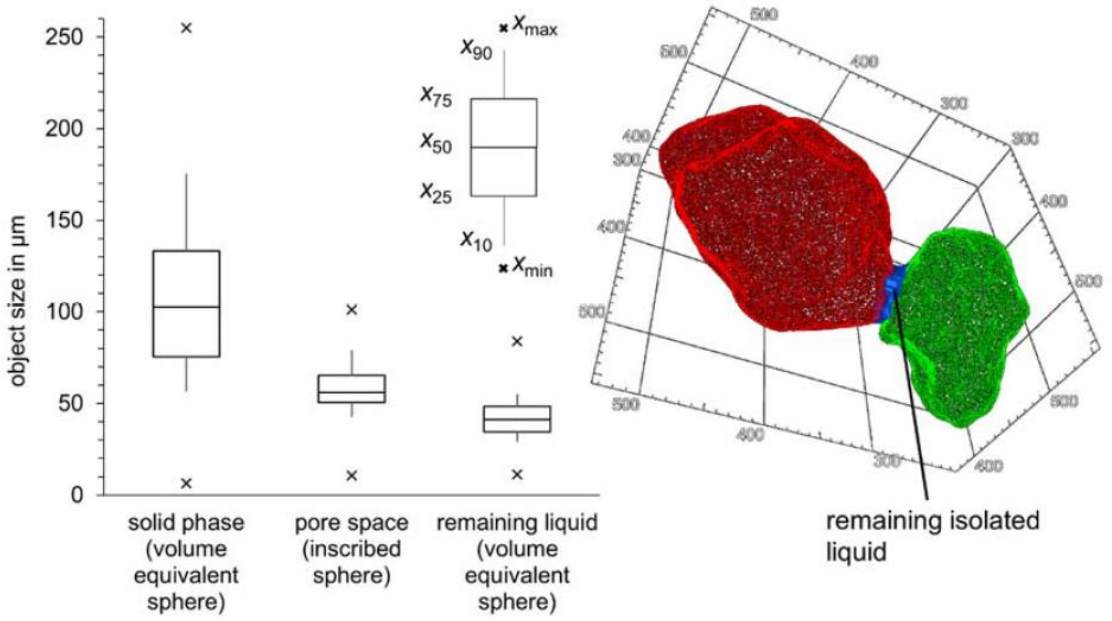
The project aims at the connection of the disperse particle properties with characteristics of the resulting filter cake structures. These include particle size, shape and wettability and the resulting particle-particle interactions.
Finally, these investigated relationships will help to better understand the relevant process properties such as the dewatering behavior or the washability of these systems.
For this purpose, mainly X-ray tomographic investigations of the filter cake structures are carried out.

Project Status:

The main scope of the last year was on finalizing the data exploitation methods. This is due to the fact, that commercial software solutions did not deliver satisfying quantitative results. Therefore, individual models and algorithms have been implemented to extract much more information out of the X-ray images.

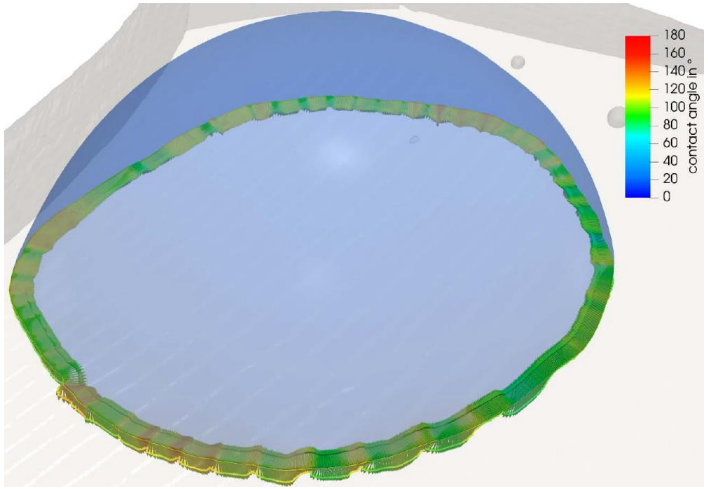
Questions & Answers

- How small can micro-CT resolve? Theoretically down to 3 micron particle size at a voxel size of 0.3 micron – but with the given filtration cell dimensions the measurement of all phases (s-l-g) requires spherical particles >40 – 50 micron.
- Do you think this method can be applied to ensembles of smaller particles? Particle sizes <20micron, having liquid bridges below 4micron require a different setup: Synchrotron- or High-Resolution-SEM is part of the research program, but goes beyond an IFPRI project.



- How do ions effect the wettability of the alumina surface (sulfates, chlorides or cations)? Sulfates may have an impact on wettability, depending on pH. For Chlorides this is not believed. No tests have been carried out. In general, the ionic strength DLVO effects are superimposed to the hydrophobic interactions.
- How does temperature play a role in these systems? The hydrophobic coating of the Alumina is only stable up to 120 degC. The impact on wettability can be investigated on demand.

Questions & Answers



- The SEM shows the sintered Alumina particles having a rather rough surface. To which reference plane is the modelled contact angle defined? The wetting angle is determined at every point of the three phase line, which results in a distribution relative to the measurable particle plane. This distribution is compared to the sessile drop distribution on a planar surface.
- The modelled capillary pressure curve appears to be a static representation of the moisture distribution. How does this compare with the equilibrium moisture in a dynamic dewatering process at constant pressure as it is typically done in the lab filter cell? Micro-CT is only able to determine static states of the dewatering process. However there is a good correlation with the equilibrium moisture content at defined pressure levels, indicating that capillary re-distribution effects are minimal.
- The decreased porosity with better wettability did not quite correlate with pore size and coordination number. Have you observed a variation of the hydrophobic agglomeration status in the suspension (sedimentation rate, sediment volume) with the wetting behavior? Hydrophobic agglomeration has been observed in another project, and sedimentation tests will be investigated.

Comments

- Over the past years an extensive amount of work has been performed to look at localized filter cake properties (structure, moisture distribution) using micro 3D-Tomography.
- The improvement of the data modelling methods took more time than anticipated, now resulting in a much better alignment with lab validation methods.
- So far, the cake structure was investigated as function of wettability. The impact on cake resistance/filterability would be of industrial interest.
- To date, extensive characterization was done for compact particles and not yet on needle-like particles, due to higher complexity. Collaboration with TU Munich is started to characterize fibers; approach may be usable for needle like particles, to be explored this year.
- The analysis of centrifugal sediment cake formation and dewatering along with filter cake washing will be subject of the renewal process in 2021.

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International Fine Particle Research Institute

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3D Printing of Perfect Particles

Prof Karen Hapgood

IFPRI AGM, May 2020

School of Engineering,
Deakin University, Australia

Dr Negin Amini

Dr Jun Zhang

Dennis Golchert (Roche)

Exploiting a Framework
for the Development of Segregation Rate Models

J. J. McCarthy

Department of Chemical Engineering
University of Pittsburgh

June, 2019

Paul Mort (Purdue)

Flowability Assessment of Weakly Consolidated Powders

Colin Hare[1], Ali Hassanpour[2], Azza Mahmoud[1], Alexandros Stavrou[1]

[1]Department of Chemical and Process Engineering, University of Surrey
[2]Institute of Particle Science & Engineering, University of Leeds

Tim Freeman (Freeman Technology)

Responses to IFPRI member questions

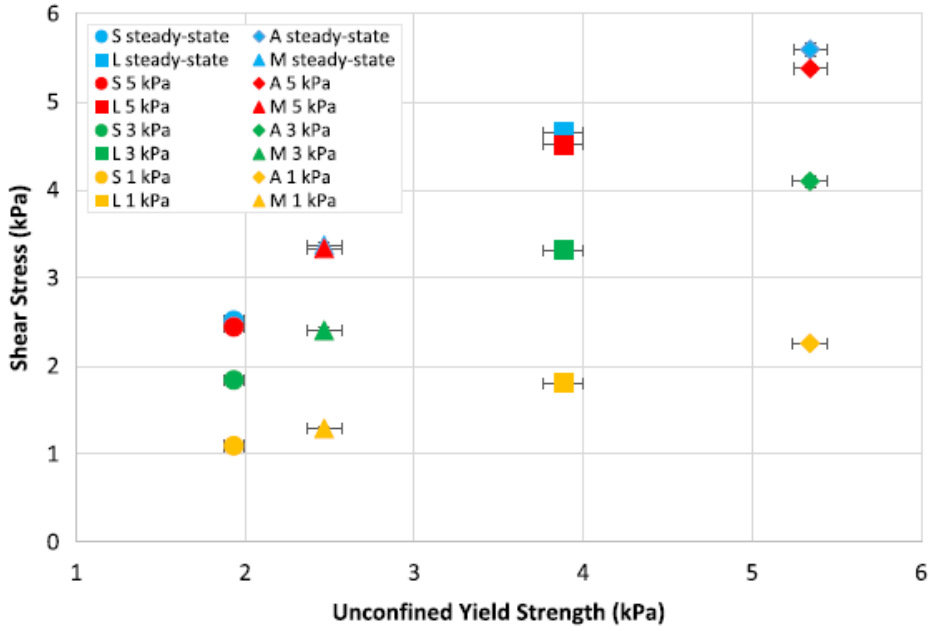
Member 1:

Precis of question:

- In experiments constraint factor is defined as $C = H/\sigma_c$
- In DEM constraint factor is defined as $C' = H/\sigma_D$
  - where σ_D is the deviatoric (shear) stress below the indenter
- Therefore, how are the DEM and experiments related

Response:

- The definition used in experiments is correct
- In DEM, simulations of uniaxial compression were unsuccessful
  - Low cohesion – bed collapsed when die walls removed
  - High cohesion – bed compressed rather than failing under shear
  - All levels of cohesion gave one of the above behaviours



From: Stavrou *et al.* (2020)
https://doi.org/10.1016/j.ces.2019.115307

- Therefore, a different failure stress was applied to obtain 'effective constraint factor', *C'*
- Experiments show that for a given pre-shear stress, shear stress is related to unconfined yield stress

- It is expected that $C' \neq C$
- However, a change in *C'* (e.g. for different frictional properties) is expected to be indicative of a relative change in *C*

Responses to IFPRI member questions

Member 2:

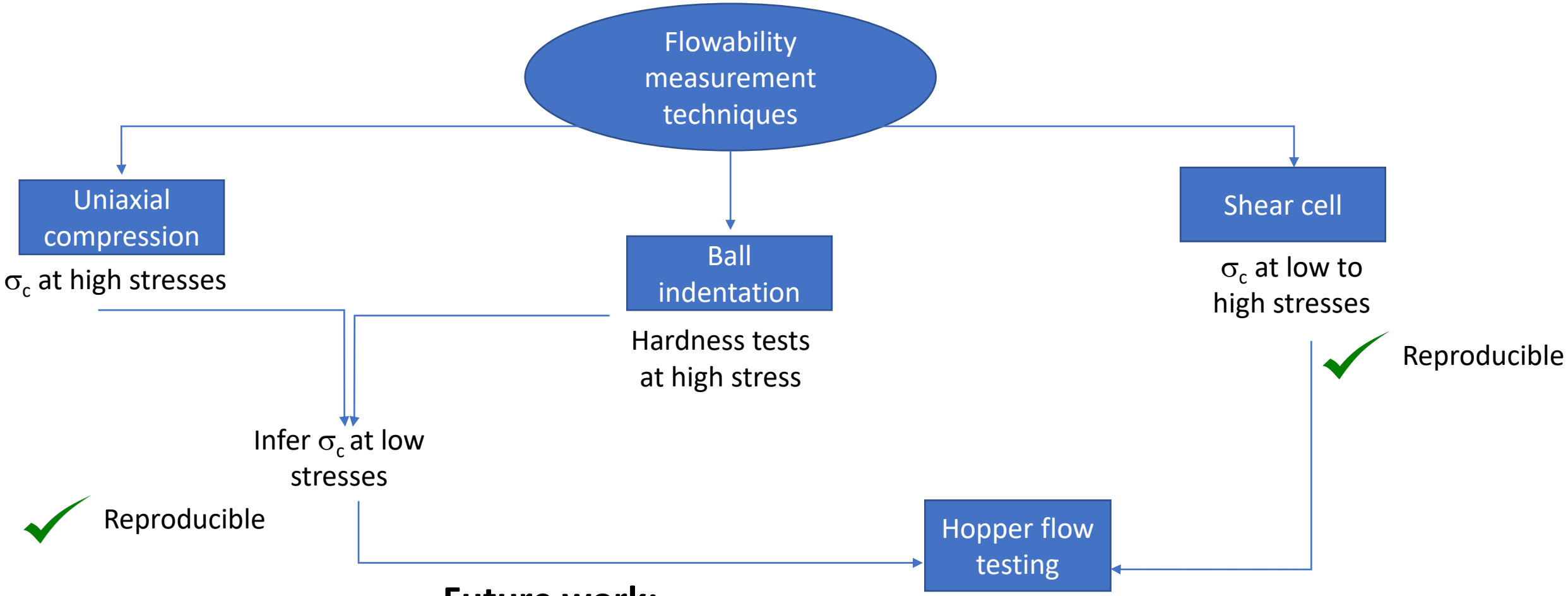
1. Does powder compressibility have an influence on penetration depth or is preconditioning and pre-shearing enough to eliminate this influence even at low stresses?
2. Have you considered to look for time dependencies – does it matter when the measurement is done after preconsitioning and pre-shearing?
3. Have all the measurements been done at controlled humidity? If so, at which level og humidity?, If not, have the humidity been recorded?
4. How many repeats are you normally doing?

Response:

1. Penetration depth is controlled, during indentation the bed should be sheared but not consolidated
   - The stable penetration depth range does vary with material
   - It is not known whether compressibility influences this
2. Delay in flow testing means the material may respond to the environment, e.g. moisture pick-up/loss
   - This may change surface properties and/or packing state, so is not indicative of the consolidation state
   - Investigating this is out of scope
3. Humidity has not been controlled but has been measured.
   - The range is 30 – 65 %RH, typically 35 – 55 %RH
4. Typically 3 repeats for shear cell tests, 5 repeats for ball indentation

Future Work

Part 1: Accuracy of flow measurement techniques



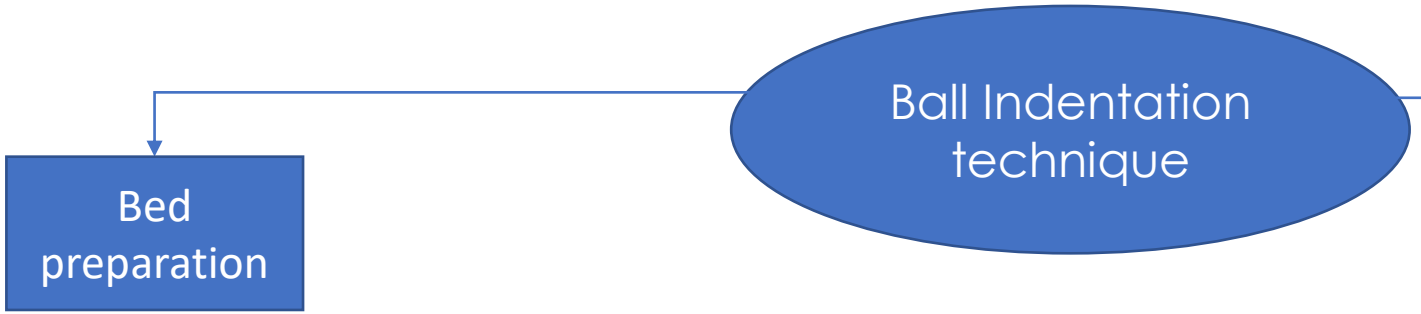
Future work:

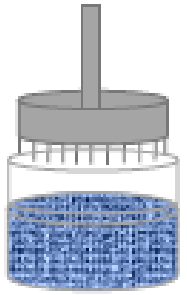
- Fabricate two separate hoppers with the determined outlet sizes (25 and 10 cm for SC and BI, respectively) – for testing Titania DT51

Or

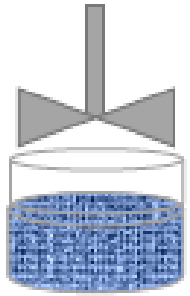
- Fabricate one hopper with a good inclination (hopper angle) allowing different outlet sizes to be tested.

Part 2: Ball indentation conditions and bed packing

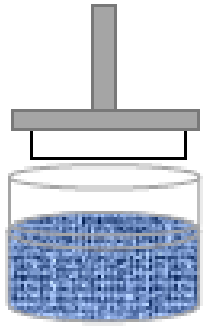




Pre-shearing



Blade conditioning



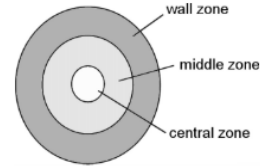
Wire conditioning

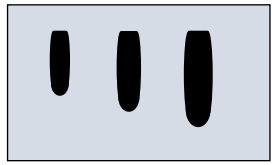
Sieve-filling

Reproducible

Indentation conditions influence on *C* and on the inferred σ_c

- Penetration **depth** (% of indenter radius)
  - Identifying stable hardness range, if it exists
  - Applying two different depths (20, and 50%)
- Indentation **position** (central, radial)
- **Indenter size** (4, 6, and 10 mm indenter diameters)





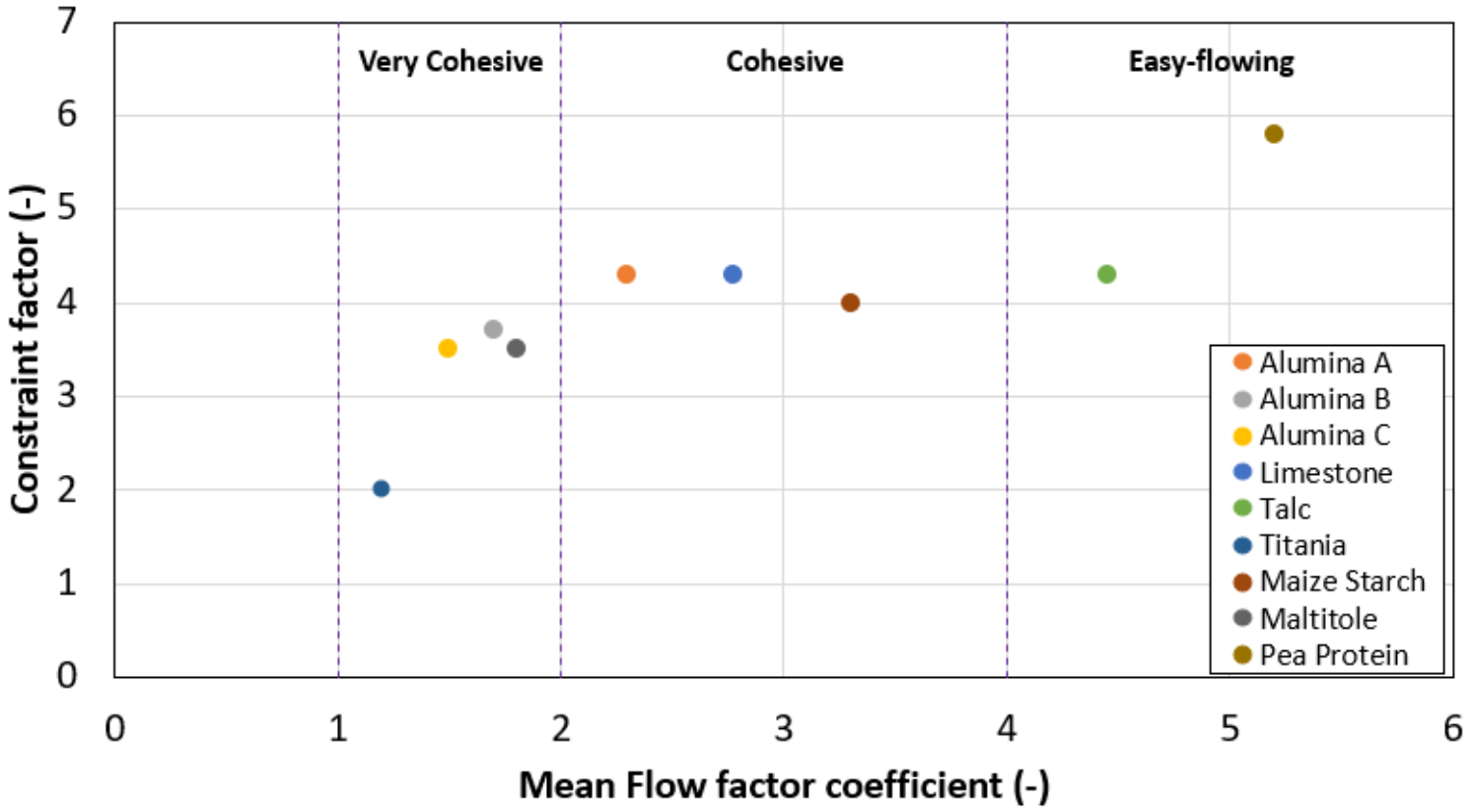
Findings:

- Stable hardness range is not always found
- Bed hardness in centre is always greater than at radial position
- Larger indenter size provides more reliable hardness measurements

Future work:

- Complete the required measurements for the study
- X-Ray tomography for two materials (ff_c ~ 1-2, 4)
  - Packing state before and after compression
  - Indentation zone after indentation
  - Indentation zone at different penetration depths (20, 50%)
  - Indentation zone at 0 and 1 kPa

Part 3: Constrain factor correlations



FF_c data Alumina **B** & **C**: at σ_{pre} of 8, 10, 12 & 14 kPa
FF_c data for all other materials : at σ_{pre} of 2, 4, 6 & 8kPa

Future work:

- Correlating *C* to flow behaviour determined by uniaxial compression tests
- Investigating correlations for *C* against loose & compressed bulk density and compressibility
- Investigate wider range of materials
  - Including Titania A1, Limestone, and Talc at same penetration depth (50%, previously 20%)

Future experimental work - summary

Accuracy of techniques - Hopper flow testing:

- Fabricate two separate hoppers with the determined outlet sizes (25 and 10 cm for SC and BI, respectively)

Or

- Fabricate one hopper with a good inclination (hopper angle) allowing different outlet sizes to be tested

Ball indentation – indentation conditions and bed packing:

- Complete the required measurements for the study (repeats)
- X-Ray tomography for one or two materials
  - Packing state of sieved loosely and compressed packed beds
  - Indentation zone after indentation
  - Indentation zone at different penetration depths (20, 50%)
  - Indentation zone at 0 and 1 kPa

Constraint factor correlations:

- Correlating C to flow behaviour determined by uniaxial compression tests
- Investigating the influence of materials loosely bulk density and compressibility on the determined C
- Investigate wider range of materials
  - Applying Uniaxial, BI and SC
  - Repeating indentation tests for the previously tested materials (Titania A1, Limestone and Talc) but at 50% dimensionless penetration depth

Environmentally Responsive "Smart" Particles

Presented: IFPRI 2020 Virtual AGM

IFPRI
International Fine Particle Research Institute

Southwest Research Institute

James Oxley, Ph.D.
Staff Scientist
James.Oxley@swri.org
Prepared May 22nd, 2020

Chris Rueb (AVEKA)

Advanced Particle Sensors LLC

particle characterization for the real world ™

Response to Questions on the Review of In-Process Characterization

David M. Scott

Advanced Particle Sensors

david.scott@particlesci.com

6/20/20

Vincenzino Vivicqua (Johnson Matthey)

Question 1 - Can measurement of small particles in the midst of large ones be achieved by combining different inline/online techniques, also depending on the solid volume fraction?

- It depends on the sizes, size ratio, and relative concentration.
- Most techniques rely on scattering, which is proportional to d^2 for coarse particles and d^6 for nanoparticles, so the fines signal for a 1:10 size ratio can be lost in the noise for intensity-only detection (e.g. DLS). The search found Photon Density Wave results for bidisperse polystyrene particles in a 1:3 size ratio (Bressel et. al 2015); a similar result for soot (not in scope) was obtained by Laser-Induced Fluorescence (Cenker et al. 2015).
- If the fine fraction is ≥5% of the solids volume and the turbidity is relatively low, laser diffraction can measure size ratios of ~1:100, because it considers angular dependence of intensity, and light from the two size classes hits different detectors. However, single fine contaminant particles generally scatter too little light to be detected. (In off-line applications, one can use filtration or centrifugation to remove large particles that obscure the fines.)

- If a wider size range or more sensitivity is needed, one might combine sensors optimized for the two size ranges, provided scattering from the coarse fraction does not prevent detection of the fines signal. An example from the search is a patent (King et al. 2016) that combines bright field and dark field microscopy to examine coarse and fine particles in the 0.05 – 500 micron range. The lower limit is not credible, but the technique can probably span a ~1:1000 size ratio. Concentration must be $<10^7$ count/mL, and thousands of images are required for PSD measurement (see ISO 13322-1 Annex A).
- Flow Cytometry uses functional fluorescent tags to detect and count fine bioparticles of interest (Sincock and Robinson 2001). Could we "light up" the fine particles in question?
- Question 1 presents a challenging technical issue best tackled for specific cases. I would be interested in working on such a project if anyone has a real-world problem to be solved.

Question 2 – Is it possible to provide some guidelines for the selection of the best combination of existing and/or emerging techniques for the in-process characterisation of wet systems?

- Some general guidance is provided on the following pages, but note that selection is case-specific: there may be several, 1, or 0 suitable techniques for any given application.
- These are suggestions to be considered – not recommendations!

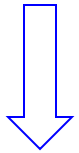
Particle Size

Technique	Size (μm)	Concentration	Size Data
On-Line Laser Diffraction	1-1000	Low-med.	Full PSD (volume basis)
FBRM*	5-1000	Low-high	Median size (with calibration) (progress on PSD extraction)
Imaging	5-1000	Low-med.	Full PSD (number basis)
Ultrasonic Spectroscopy	0.1-500	Low-high, opaque systems	Size & spread
Spatially Resolved DLS*	0.01-3	Low - Med. (turbid)	Size & spread
Acoustic emission*	>150	High	% oversize

* Technique mentioned in the review

Fractal Dimension

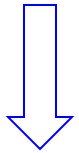
- Image analysis *
- Small-angle light scattering *



Increasing complexity

Shape & Aspect Ratio

- Image analysis *
- Laser diffraction *
- Parsum probe *
- Dynamic Light Scattering *
- Photon Density Wave spectroscopy *



Increasing complexity

* Technique mentioned in the review

Gas Holdup & Phase Distribution

- Electrical Resistance Tomography linear probe* (for conductive systems)
- Electrical Capacitance Tomography (for non-conductive systems)
- Ultrasonic backscatter* or through-transmission profiling

* Technique mentioned in the review

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- ISO 13322-1:2014 Particle size analysis — Image analysis methods — Part 1: Static image analysis methods

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DEM Round Robin: Toward a Best Practice for Powder Flow Simulation

Dr. Kit Windows-Yule
School of Chemical Engineering
The University of Birmingham

Massih Pasha (Chemours)

IFPRI DEM Round-Robin

University of Birmingham's Progress

Material 1: MCC				
Material acquisition	*Material dissemination*	*PEPT Drum*	*PEPT Mixer*	*Lenterra experiments*
completed	in progress	completed	completed	completed

Material 2: Angular sand				
Material acquisition	*Material dissemination*	*PEPT Drum*	*PEPT Mixer*	*Lenterra experiments*
completed	in progress	completed	completed	

Material 3: Ion exchange resins				
Material acquisition	*Material dissemination*	*PEPT Drum*	*PEPT Mixer*	*Lenterra experiments*
completed	in progress			

DEM Participant's Progress

Participants	Material 1: MCC			Material 2: Angular sand			Material 3: Ion exchange resins		
	Experimental Characterisation	*Simulation High Shear Mixer*	*Simulation Rotating Drum*	*Experimental Characterisation*	*Simulation High Shear Mixer*	*Simulation Rotating Drum*	*Experimental Characterisation*	*Simulation High Shear Mixer*	*Simulation Rotating Drum*
AbbVie	completed	completed							
Chemours	in progress								
DFE**	in progress								
DSM	completed	completed	completed				in progress		
Merck	in progress								
P&G	in progress	in progress	in progress						
Sandia	in progress	in progress	in progress						

Characterization Participant's Progress

	Material 1: MCC	Material 2: Angular sand	Material 3: Ion exchange resins
Freeman Technology	completed	completed	completed
Granutools	completed	completed	completed