Relating Bulk Powder Flow Behavior to Powder Properties

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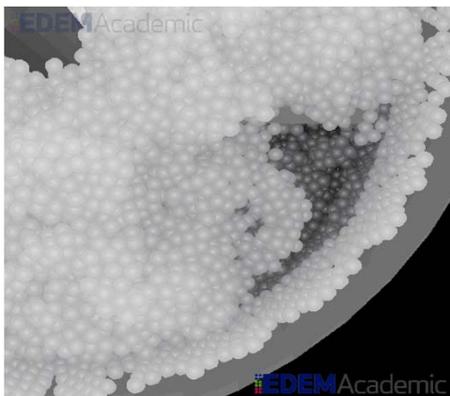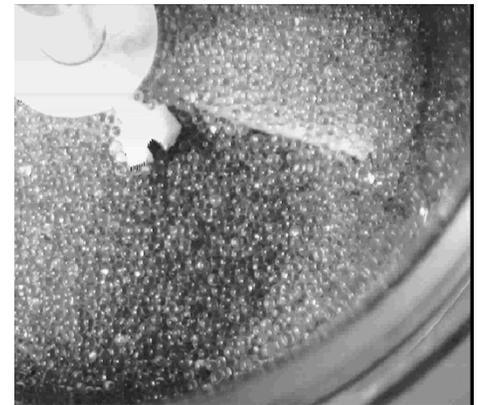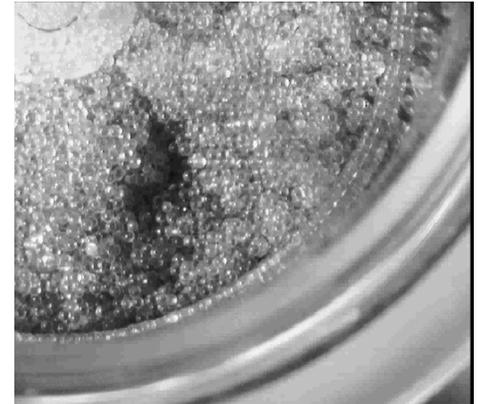
Particle properties and bulk flow: grand challenge

- In manufacturing, one would like to take a small number of measurements with a small amount of powder using inexpensive bench scale equipment and use this information to predict bulk flow behavior at the manufacturing scale equipment.

Image source: www.secretsensations.org

- Jenike (1961, 1964) does this for flow of a powder through a hopper. You measure a few properties of the powder and use theory and supporting equations to predict flow out of the hopper. Works in many cases – doesn't work when powders cake, time-consolidate, etc.

- For feeders and many other operations, this usually doesn't work i.e. we have not found a set of powder properties that correlate to bulk flow behavior → trial and error, informed design based on previous experience: do a large number of measurements with a relatively large amount of powder on bench scale and pilot scale, and hope to predict manufacturing scale.

- Flow, Segregation and Mixing of Particles

- Heat and Mass Transfer in Particles

  – Drying of pharmaceutical crystals, drying of catalyst pellets, rotary calcination
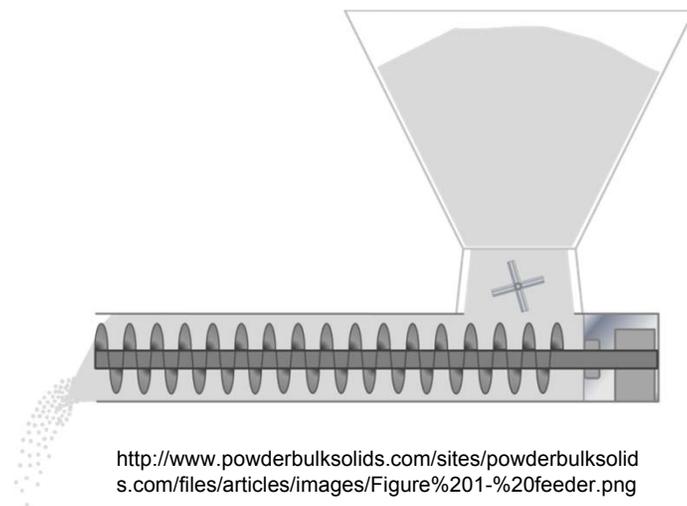
- Fluidized Beds, Gas-Particle Flows
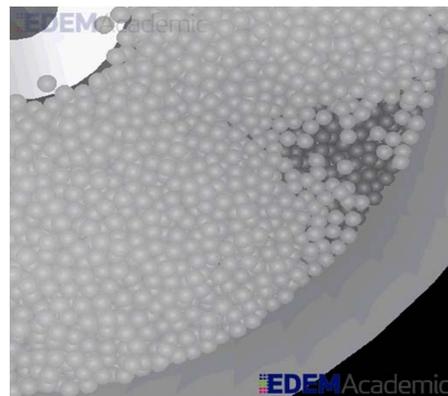
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- Fluidized Beds, Gas-Particle Flows
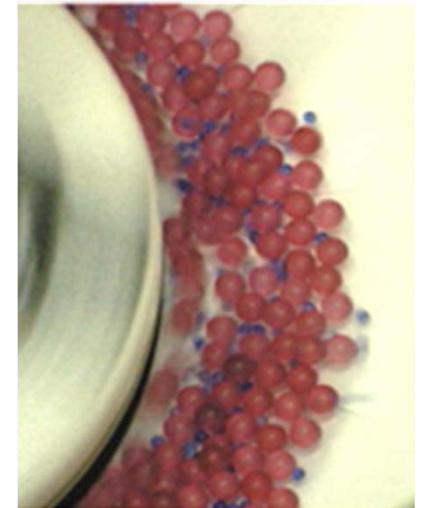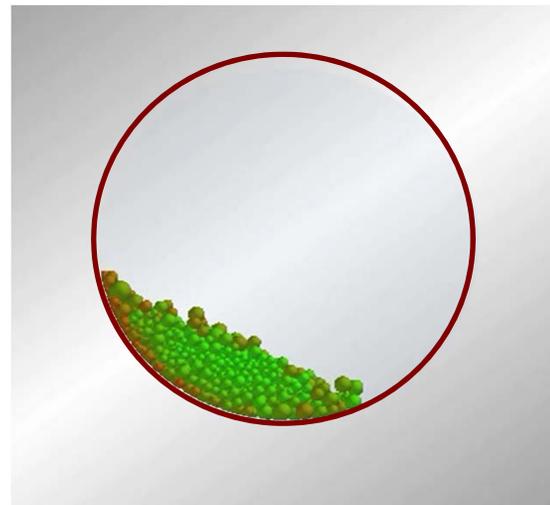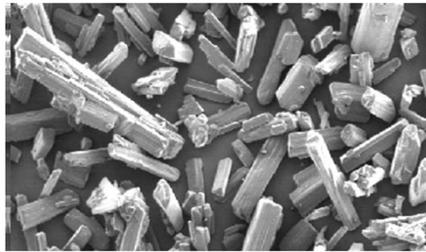
Particle properties and bulk flow

- Can we use statistical methods to correlate powder properties to flow behavior?

- In pharmaceutical clinical trials and development, we don't expect to have an equation or theory; we correlate clinical outcomes with drug administration using statistical methods.

- Current work: use statistical methods to correlate feeder performance to powder properties.

With: Yifan Wang, Tianyi Li, and Fernando Muzzio

http://www.powderbulksolids.com/sites/powderbulksolids.com/files/articles/images/Figure%201-%20feeder.png

5

Continuous Manufacturing of Pharmaceuticals: Rutgers Direct Compression Line

Feeders

API

EX

Mill

Blender

Tablet Press

Also potentially for catalyst manufacturing
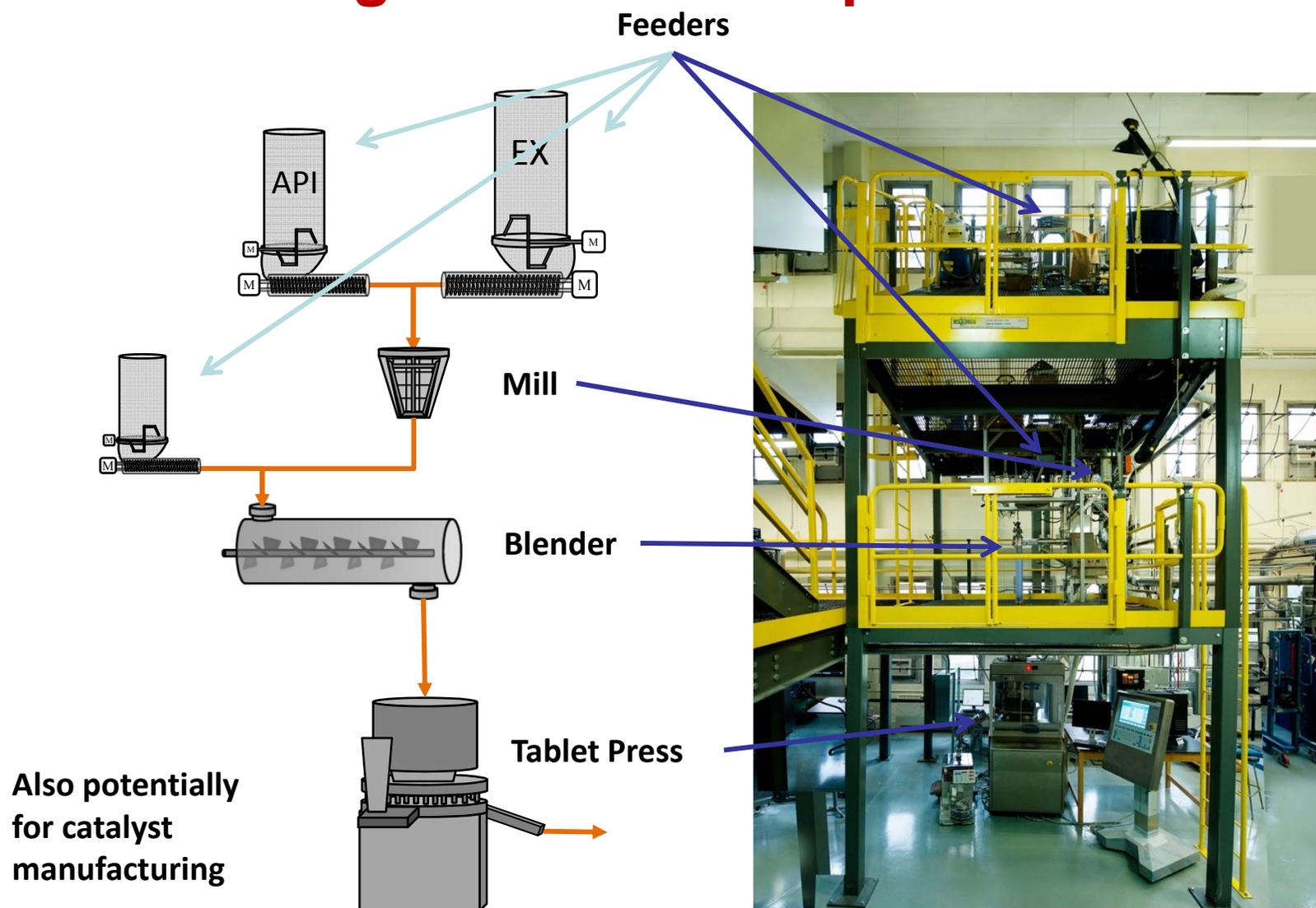
Predicting Feeder Performance

- 30 flow variables from 10 characterization techniques using 2 devices
- Particle size: Beckmann-Coulter Laser-diffraction (LS-13320)
- Material flow properties: Freeman Technology FT4 Powder Rheometer, Freeman Technology Inc.

  d10, d50, d90, Cohesion, Unconfined Yield Strength, Major Principal Stress, Flow Function Coefficient, and Angle of Internal Friction at initial consolidation stresses of 3kPa, 6kPa, 9kPa, and 15kPa. Conditioned bulk density, Compressibility index, Pressure drop, Basic Flow Energy, Stability Index, Specific Energy, Flow Rate Index

  3 replicates, 100g of powder, 4hrs

- (Currently adding Electrostatics: dry and wet impedance, developed in-house)

Predicting Feeder Performance

- **Correlation of powder properties to feeder performance:**
- Develop methods to predict feeder performance based on material properties
- Implement material library using existing materials
- Build up a multivariate model to predict feeder's performance based on material flow properties
   i. PCA-SS (Principle component analysis followed by similarity scores)
   ii. PLSR (Partial least squares (PLS) regression)
- **Variable reduction:**
- What is the minimum measurement set to identify a material?

Image source: wholesaler.alibaba.com

Feeder Performance Evaluation

- Material properties have a significant effect on feeder performance.
- A loss-in-weight feeder consists of three parts: volumetric feeder, weighing platform, and gravimetric controller.
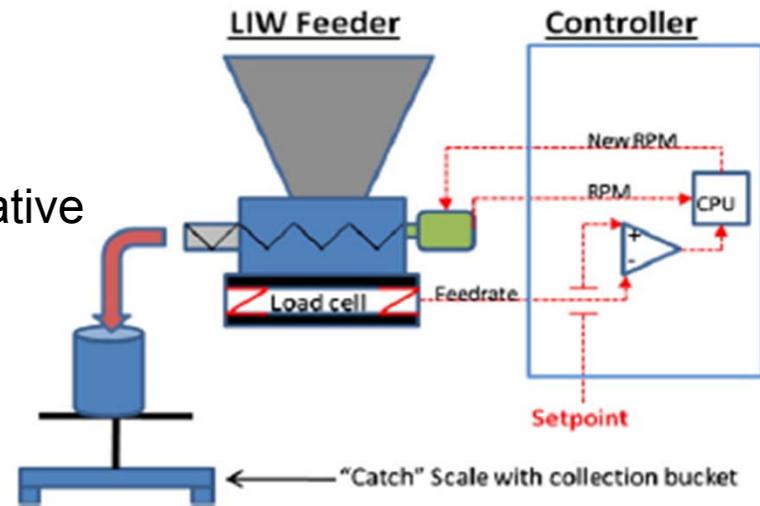- The target mass feed rate is set at *m*, the actual mass feed rate is calculated by

$$\dot{m} = \frac{\Delta m_i}{\Delta t}$$

- Relative standard deviation (RSD) and relative difference from the mean (RDM) is used as criteria for feeder performance:
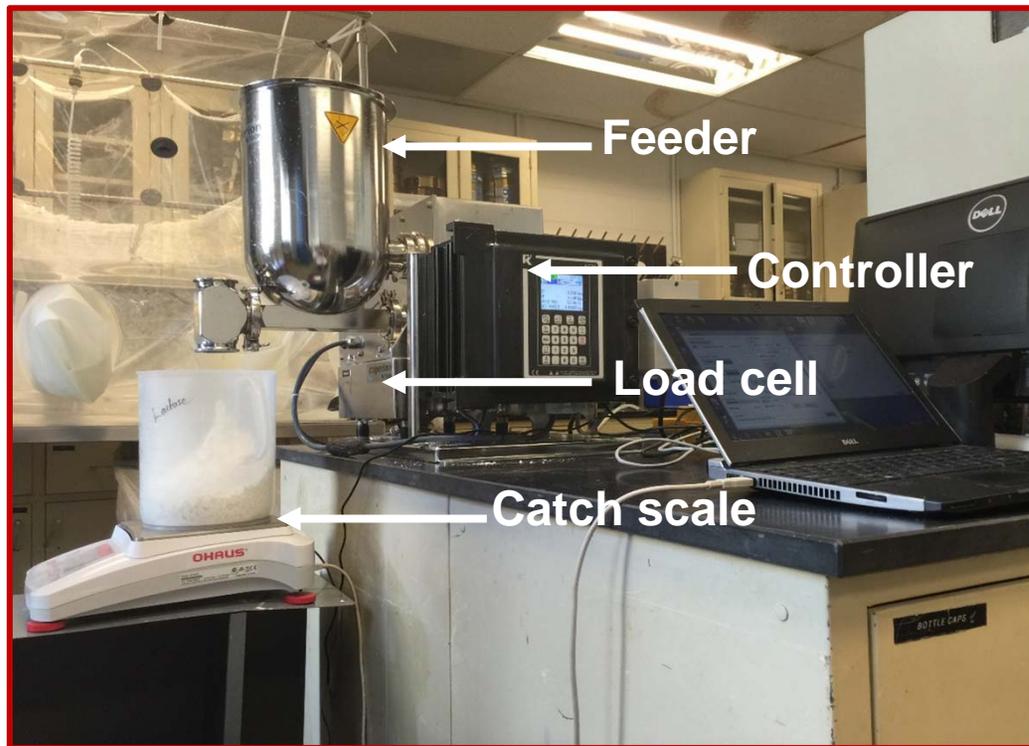
$$\sigma = \sqrt{\frac{\sum_{i=1}^{n}(\dot{m}_i - \overline{\dot{m}})}{n-1}}$$

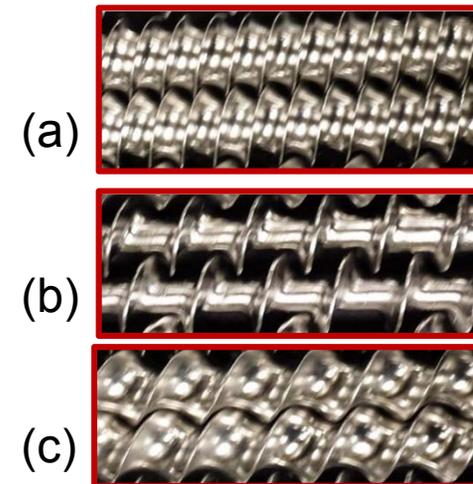$$RSD = \frac{\sigma}{\overline{\dot{m}}} \qquad RDM = \frac{|\dot{m} - m|}{\dot{m}}$$

σ is standard deviation, $\overline{\dot{m}}$ is the arithmetic mean of the actual mass feed rate, and n is the number time points.

Reference: Engisch, William E., and Fernando J. Muzzio. "Method for characterization of loss-in-weight feeder equipment," *Powder technology,* 228, (2012): 395-403.

Loss-in-weight feeder experimental set-up

K-Tron Feeder

Experimental set-up of characterizing feeding performance for each material. Available screws for feeder experiment: (a) screw type 1, fine concave screw, (b) screw type 2, fine auger screw, and (c)screw type 3, coarse concave screw.

Predicting feeding performance from material flow properties

- For a given new material, can we compare it to existing materials in the library based on material properties?

- Once a new material is included in the material library, can we predict its feeder performance using a certain screw?

- Can we predict the optimal screw choice for a given new material?

Image source: www.secretsensations.org

Principal Component Analysis (PCA)

With so many powder properties, it's important to have a method to reduce the number of variables

- *PCA uses a transformation to convert the set of measurements into a set of values of linearly uncorrelated variables called principal components*
- *The direction of the first principal component (PC1) points in the direction of the maximum variation.*

Property 3
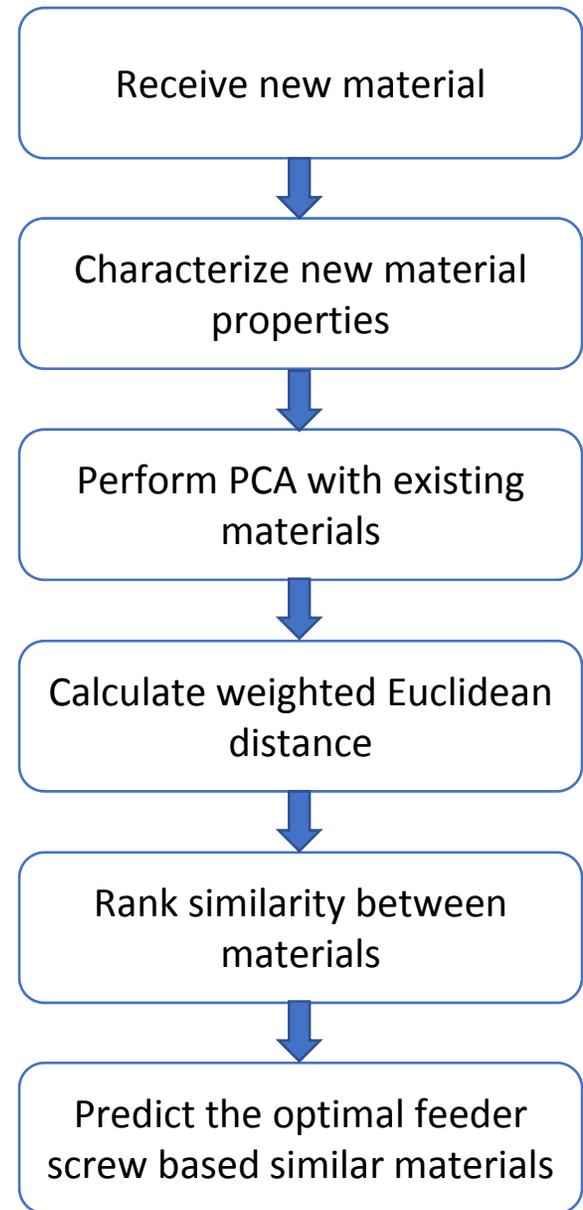
PC1

PC2

Property 1

Property 2
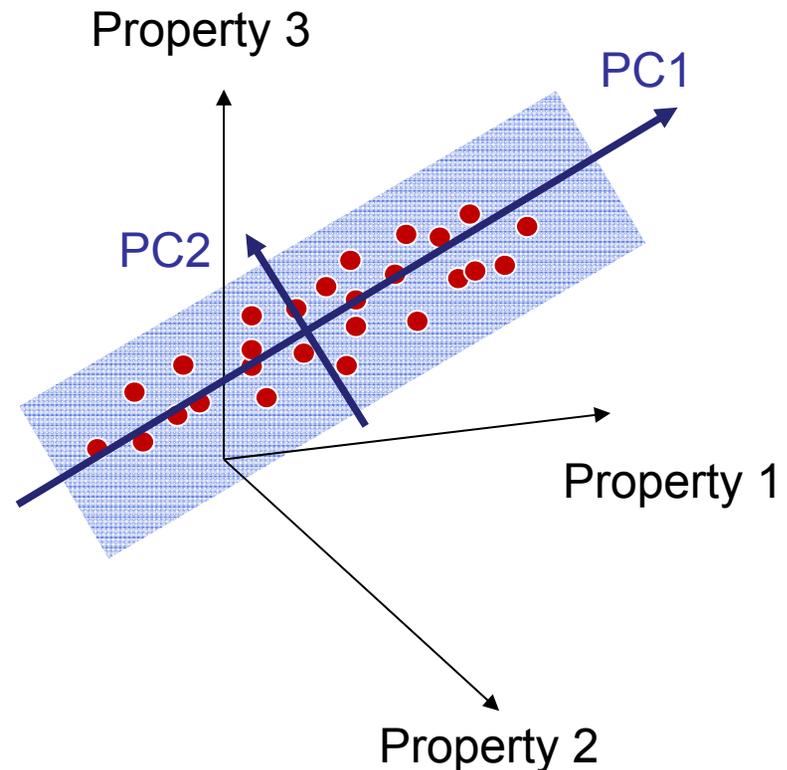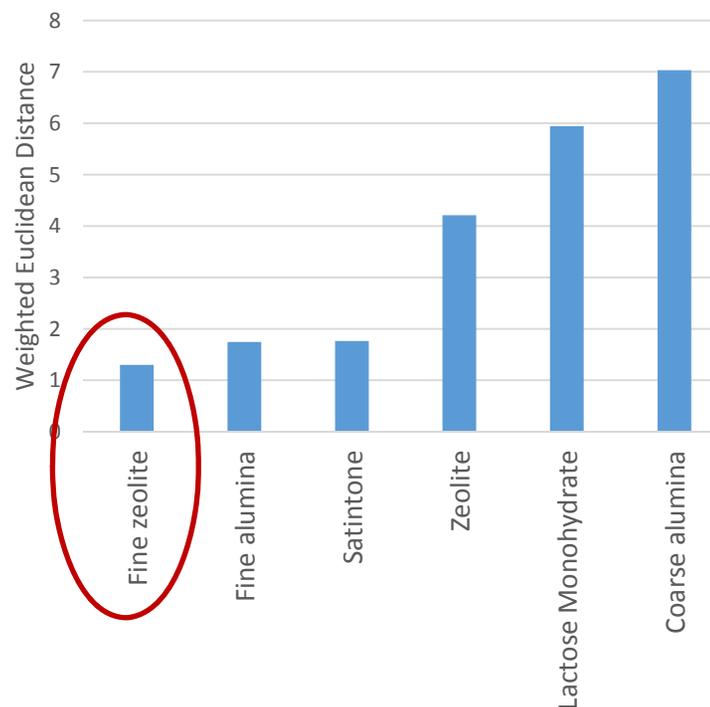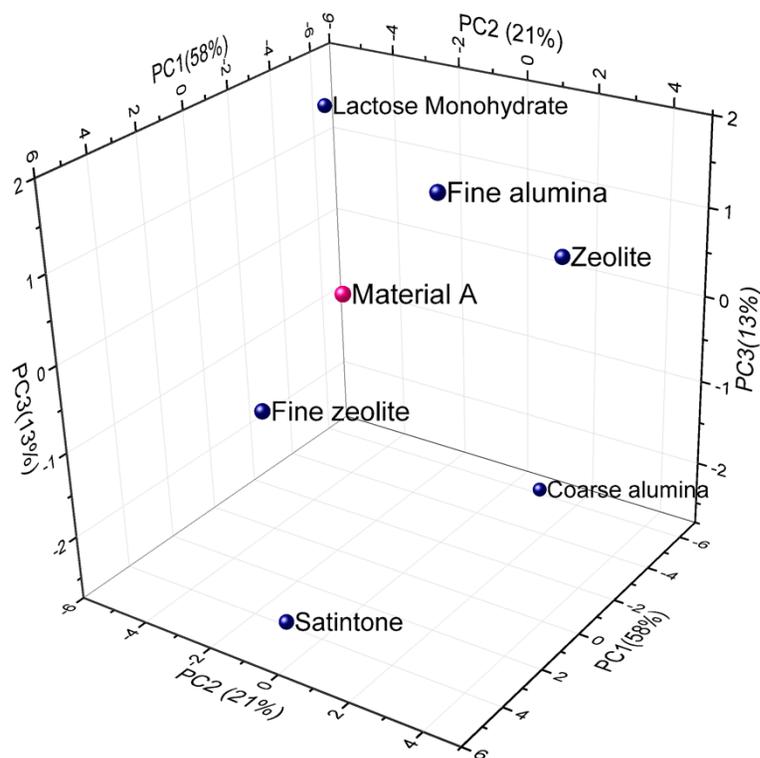
New material

Material similarity can be quantified by calculating weighted Euclidean distance. Smaller distance corresponds to higher similarity.

$$d_{w\,(a,b)} = \sqrt{\sum_{i=1}^{n} w_i\,(a_i - b_i)^2}$$

n is the total principal components selected in the model,
a_i is the score of material a in the ith principal component,
b_i is the score of material b in the ith principal component,
w_i is the relative variability explained by the ith PC

Prediction using similarity scores

screw type 1
fine concave screw

screw type 2
fine auger screw

screw type 3
coarse concave screw

Receive new material

Characterize new material properties

Perform PCA with existing materials

Calculate weighted Euclidean distance

Rank similarity between materials

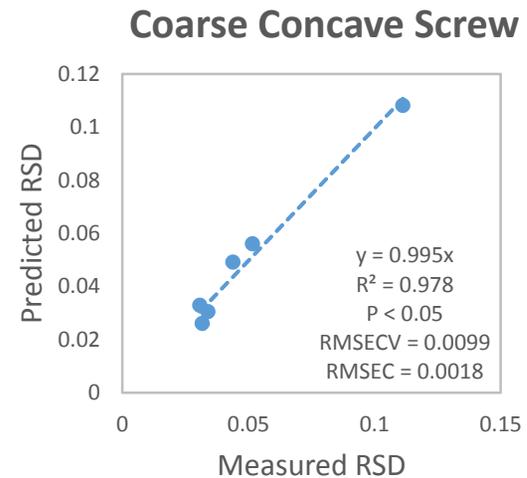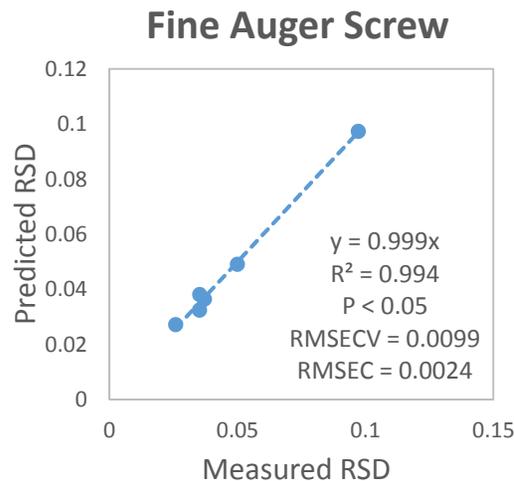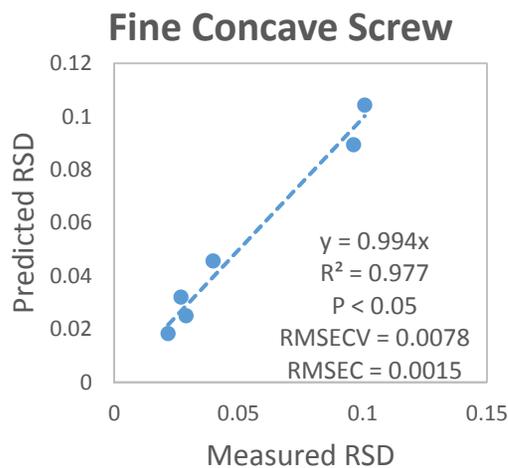Predict the optimal feeder screw based similar materials

14
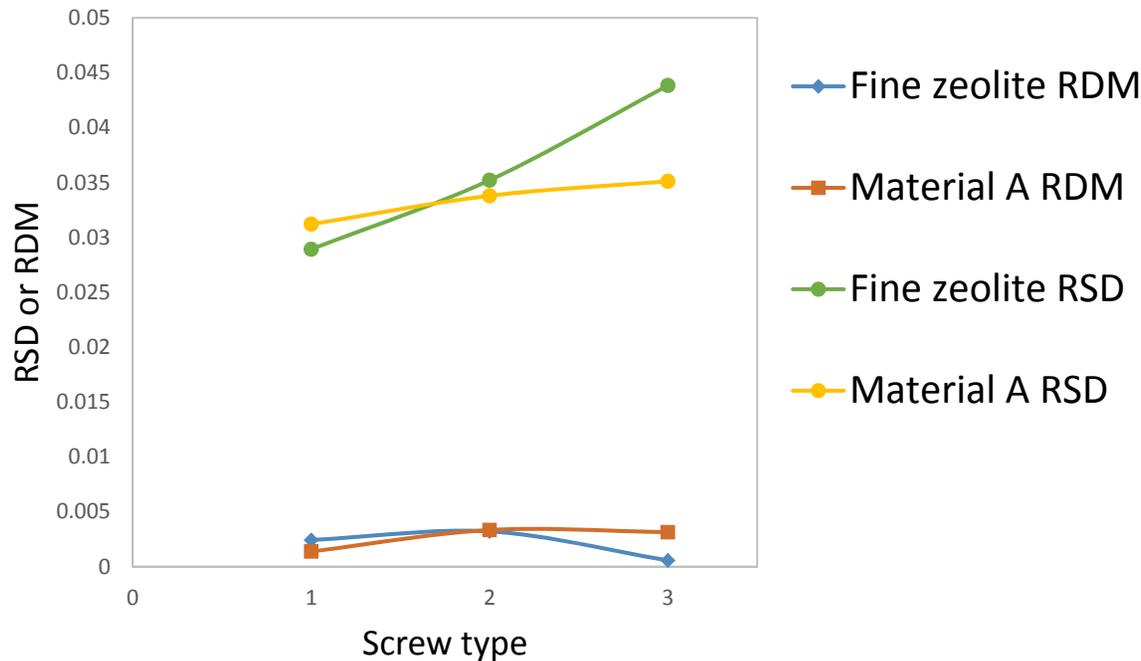
Prediction using PLS regression

Alternatively, when material with matching flow properties is difficult to find, a partial least squares (PLS) regression can be used. A PLS regression model relates material flow properties directly to feeder performance, quantified by RSD and RDM.

15
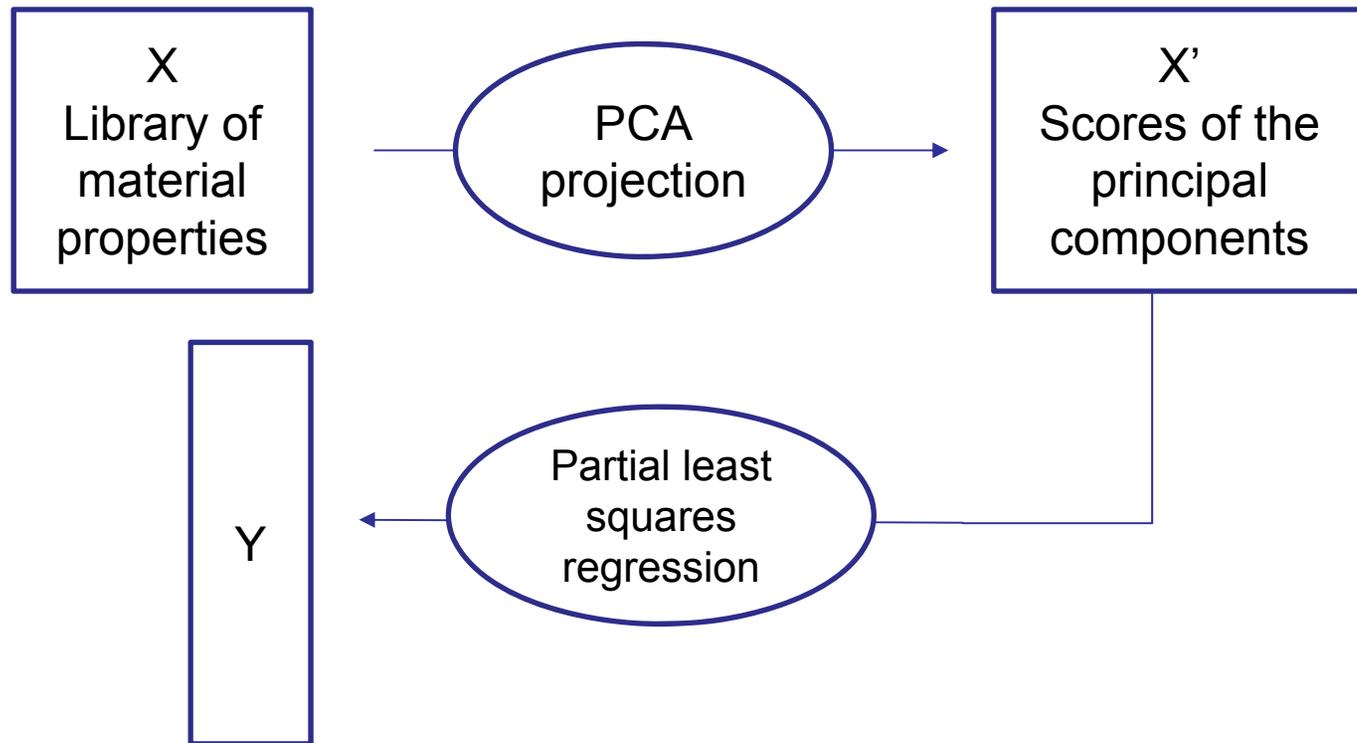
Predicting feeding performance

- PLS regression helps to answer:

1. For a new material with given properties, can we predict RSD or RDM for a certain screw?

2. For a new material, what is the optimal screw selection?

Fine Concave Screw

$y = 0.994x$
$R^2 = 0.977$
$P < 0.05$
RMSECV = 0.0078
RMSEC = 0.0015

Fine Auger Screw

$y = 0.999x$
$R^2 = 0.994$
$P < 0.05$
RMSECV = 0.0099
RMSEC = 0.0024

Coarse Concave Screw

$y = 0.995x$
$R^2 = 0.978$
$P < 0.05$
RMSECV = 0.0099
RMSEC = 0.0018

Wang, Y., Li, T., Muzzio, F.J., Glasser, B.J., *Powder Technology*, **308**, 135-148, (2017).

Prediction using PLSR models
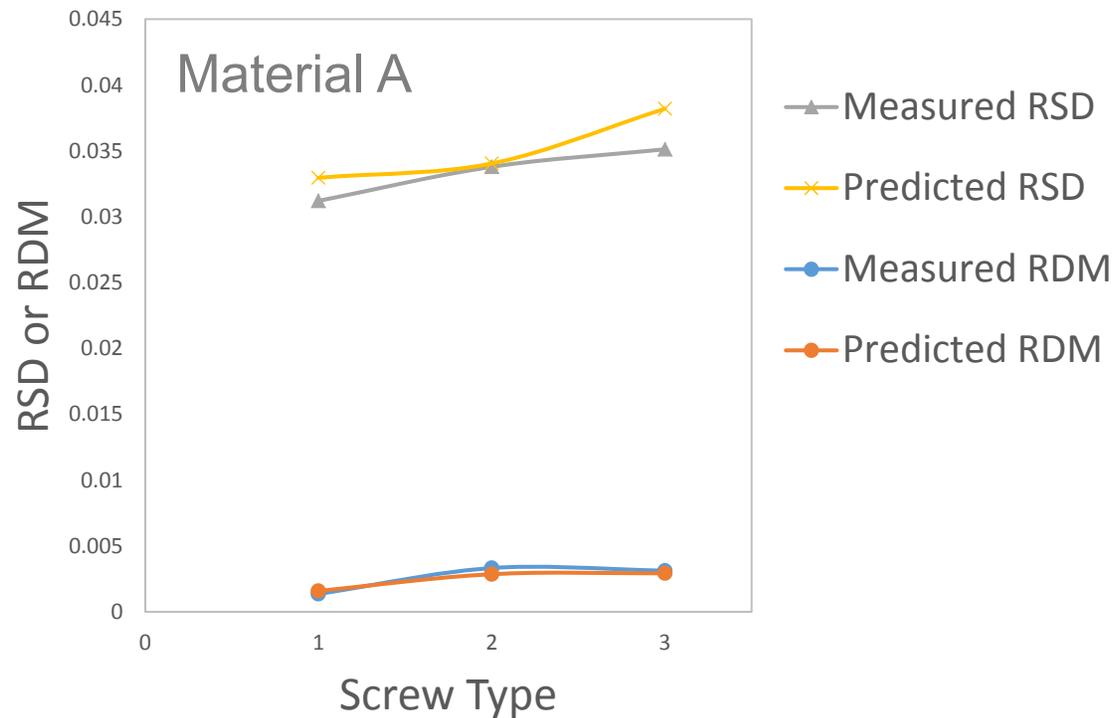
Material A

- Measured RSD
- Predicted RSD
- Measured RDM
- Predicted RDM

RSD or RDM (y-axis, 0 to 0.045)

Screw Type (x-axis, 0 to 3)

screw type 1
fine concave screw

screw type 2
fine auger screw

screw type 3
coarse concave screw

Receive new material

↓

Characterize new material properties

↓

Project data to PLSR models to predict feeder performance

↓

Select optimal screw based on model prediction

↓

Run confirmatory feeder experiment

↓

Add confirmatory data and update model knowledge

17
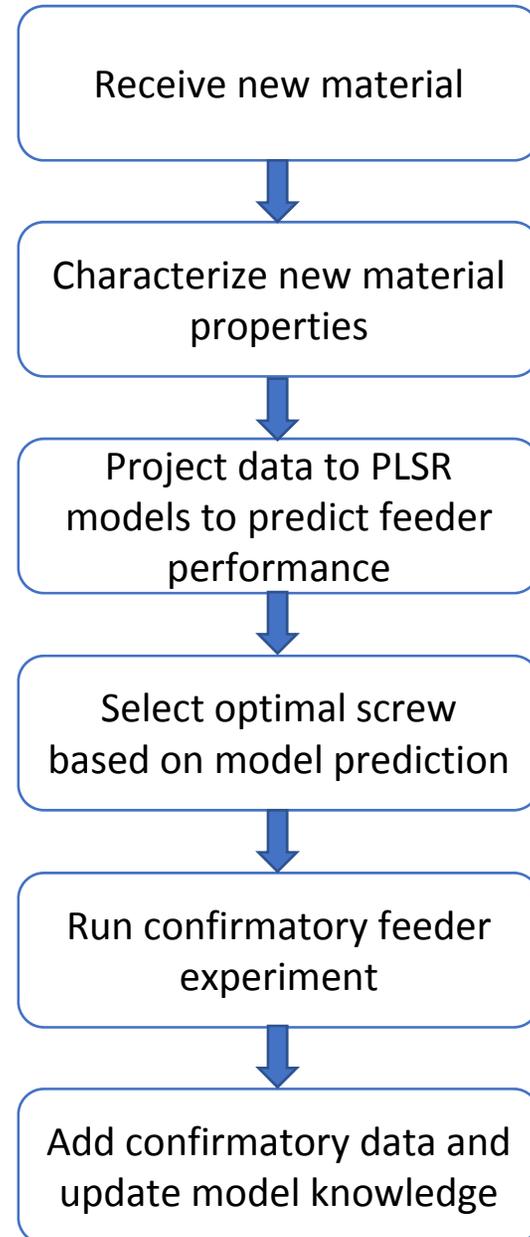
Predicting feed factor from material properties
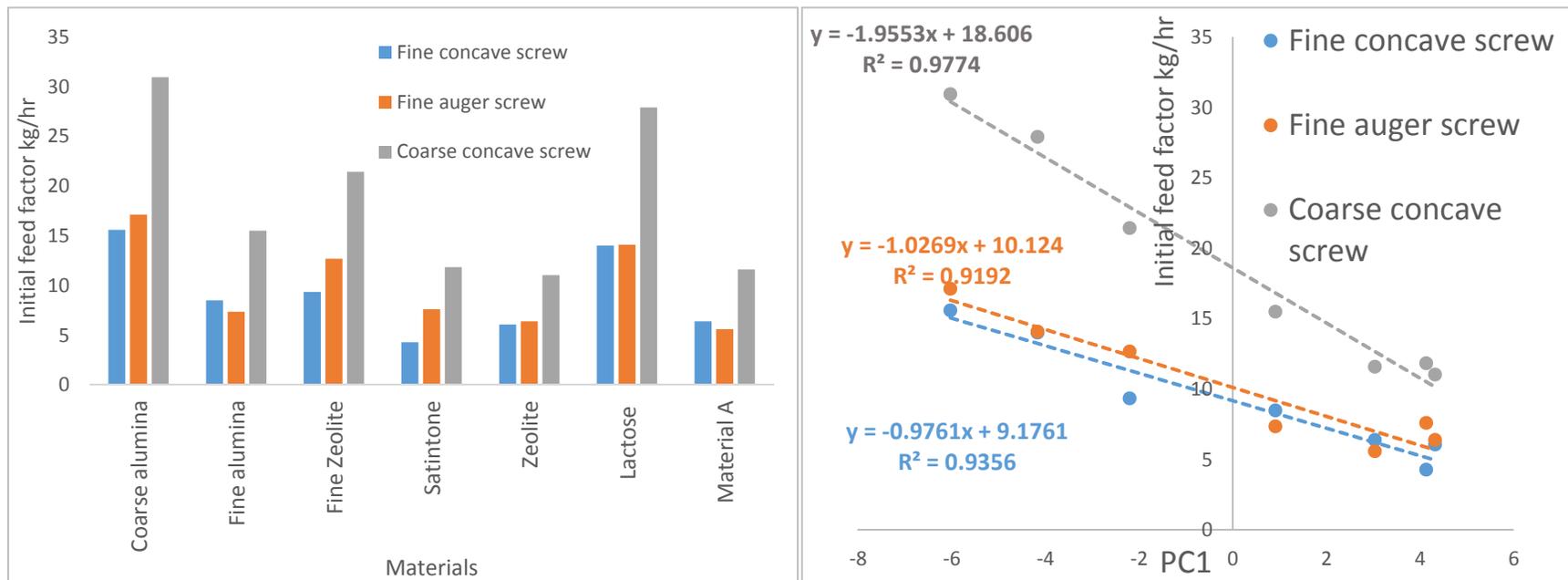
- Initial feed factor reflects maximum feeding capacity for a material.
- Results show that using scores of the first principal component, the initial feed factor can be predicted based on a linear correlation.
- The feed factor using different screws can also be predicted.

Scientific questions

- Can statistical methods be used to correlate powder properties to bulk flow behavior?

- What is the minimum set of measurements that can be used to characterize a powder in order to predict bulk flow behavior?

- Can we carry out simulations that can predict bulk powder flow behavior; discrete or continuum. What are the limitations?

Image source: www.secretsensations.org