Predicting Feeder Performance Based on Material Flow Properties

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Abstract

Purpose: Accurate and consistent delivery of materials by well-designed feeders ensures overall process stability. Importantly, feeding performance is strongly dependent on material flow properties. The purpose of this study is to develop a methodology that identifies predictive correlation between material flow properties and feeder performance. **Method:** The proposed methodology includes techniques to characterize material flow properties, methods to quantify feeding performance of a loss-in-weight feeder, and predictive multivariate analysis. Two approaches to correlate feeding performance and material flow properties were examined in the study: principal component analysis, followed by similarity scoring (PCA-SS), and partial least squares regression (PLSR). **Results:** Experimental results showed that selection of the optimal feeder screw to achieve optimum feeding performance is heavily dependent on material flow properties. Both approaches to predict feeding performance based on material properties were validated. In addition, a strong correlation between the initial feed factor of each material and its flow properties were observed. **Conclusion:** The work presented here has demonstrated an efficient approach to correlate material properties with gravimetric feeder performance. This approach is especially powerful in the early phase of process and product development, when the amount of a material is limited.

Keywords: Powder, Powder flow, loss-in-weight feeder, Multivariate analysis, Process development.

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1. Introduction:

Continuous manufacturing has been widely adopted across many industries, such as oil refining, natural gas processing and bulk chemical production [1, 2]. There are many advantages of continuous processing, including smaller scale equipment, higher flexibility, enhanced controllability, and reduced labor requirements. Studies have shown that continuous manufacturing can significantly decrease production costs while improving product quality [3]. In continuous processing, input raw materials and energy are fed into the system at a constant rate, and at the same time, a constant extraction of output products is achieved. The process performance is heavily dependent on stability of the material flowrate. For powder-based continuous processes, it is critical to feed powders consistently and accurately into subsequent unit operations of the process line, as feeding is typically the first unit operation. Feeder consistency also has an impact on mixing efficiency [4]. In particular, when multiple ingredients are fed to the process, inability to maintain constant material concentrations in the process stream can potentially lead to product failure [5]. Therefore, accurate gravimetric feeder performance has been recognized as crucial to success of the overall process.

Feeders have been designed to achieve performance reliability, feed rate accuracy, and minimal disturbances [6-9]. Loss-in-weight feeders control material dispensing by weight at a precise rate, and are often selected to minimize the flowrate variability that is caused by change of fill level and material bulk density [10]. Engisch *et al.* proposed a method to characterize and evaluate the steady state performance of a loss-in-weight feeder for different powders [11]. Methods to measure feeding performance during hopper refill and to optimize scheduling of refilling have also been presented [12]. Kehlenbeck *et al.* showed improved feeder dosing consistency by using

a single proportioning device at the discharge [13]. Tardos *et al.* used vibratory hopper agitation to improve overall flow and feeding precision [14].

In general, a particular feeder cannot handle all materials [15]. For a certain feeder, the feeding performance depends not only on equipment design and control systems and target flow rate, but also on material properties, specifically, material flow properties. For example, cohesive powders can adhere to screws, or can bridge across feeder hopper, causing phenomena called rat holes”. Free flowing materials, on the other hand, often have higher densities and thus requires more energy for the feeder to displace the materials. They can also “flush” through the feeder, causing pulsating flow rates. A poorly paired powder-feeder combination typically leads to powder sticking to equipment walls, and high feed rate variability [16]. Therefore, the selection of an appropriate feeder and feeder tooling for a powder material usually starts with characterizing the powder flow properties. In fact, some flow property measurements were initially developed to bring a mathematical approach for powder handling equipment design, for example using shear cell tests for hopper specification [17]. However, currently, feeder tooling selection for a material to achieve good feeding performance heavily relies on empirical knowledge.

Raw material properties often have substantial impact on the performance of the manufacturing process [18-20]. Identifying the relationship between raw material properties and process performance is critical for determining the effect of material property variations on downstream processes [21]. However, there is still a lack of published work on using a systematic approach to predict feeder performance based on material flow properties. Owen *et al.* demonstrated use of the Discrete Element Method (DEM) to predict screw conveyor performance as a function of operating conditions [22]. Unfortunately, material properties are not considered in the model.

Freeman *et al.* used a dynamic powder characterization test to correlate two dynamic properties to the volumetric flow rate of different screw feeders [23]. Importantly, the “flowability”(i.e., flow-related behavior) of a powder is a multi-dimensional characteristic [24]. Relying on a single characterizing technique to correlate to process performance is not sufficient [25]. Questions remain regarding which measurement or measurements provide the best predictive capability for feeder performance.

The purpose of this paper is to correlate feeder performance with material flow properties using a multivariate approach. Particularly, the aim is to answer the following three questions: i) How can we compare a given new material to previously characterized materials? ii) For a material with given properties, can we predict its feeder performance? iii) Furthermore, for a material with given properties, can we predict the optimal tooling that achieves a specified feeding performance?

It is useful to answer the above questions in powder handling and processing in industry. For example, in the early stages of pharmaceutical drug development, the amount of active pharmaceutical ingredient (API) available for testing is often limited to a 0.2-1.0 kg due to the cost of material, availability, and safety concerns [26]. Therefore, an approach that uses as little material as possible is desirable to save resources and time, and to improve efficiency of dosage form development. In addition, selection of surrogate materials that exhibit similar properties (and act as a placebo) can help to accelerate the product development process [27]. Selection of a surrogate material requires the use of prior knowledge of material properties and multivariate analysis tools to explore data structure and patterns that are often hidden through univariate methods. Additionally, the wealth of information of prior knowledge of the identified surrogate material enables one to quickly identify the design space and avoid failure modes when rapid

process design and development is needed [28, 29]. The development of pharmaceutical generic products, for example, has been facing severe pressure to lower costs, minimize waste, and accelerate development cycle. It is thus advantageous to examine the potential of using data-driven models to connect material properties with process performance, to accelerate process development for faster commercialization [30]. The data-driven and data-enriched approach used in this study is consistent with the quality-by-design (QbD) initiative by the U.S. FDA, where multivariate analysis, as an important process analytical technology (PAT) tool, was used to design, analyze and control manufacturing processes [31].

It is already known that highly flowable materials will produce better feeder performance than poorly flowing materials. However, the goal of our work is to go beyond a qualitative assessment, and make a quantitative prediction in terms of the optimal tooling for a particular material and also, quantitatively predict the fluctuations in flow rate and deviations from the target flow rate. The method to be presented in this paper includes characterization of material flow properties, characterization of feeder performance, and predictive multivariate analysis. As there is no unifying framework to describe powder flow behavior, materials were characterized by multiple flow property measurements so that each reflects a different aspect of flow behavior. To characterize feeder performance, volumetric studies were initially performed to determine feeder capability, followed by gravimetric studies to evaluate overall performance. For multivariate analysis, two approaches were carried out. Principal component analysis (PCA) was firstly used to project material properties onto reduced dimensions; the predictive capability can then be achieved by calculating similarity scores to find materials with similar flow behavior. Secondly, partial least squares regression (PLSR) was used to directly predict feeder performance based on material property inputs. The method was demonstrated by using a commercially available

feeder, a K-Tron KT20 loss-in-weight feeder, for seven powder materials that were characterized by four material flow characterization techniques, represented by 30 flow indices.

2. Materials and methods

2.1 Materials

The materials that were characterized in this study were: Coarse γ -alumina (Albermarle Inc., Amsterdam, the Netherlands), Fine γ -alumina (Albermarle Inc. , Amsterdam, the Netherlands), Fine zeolite (Albermarle Inc., Amsterdam, the Netherlands), Zeolite (Y-type CBV 100, BASF Corporation, NJ, USA), Satintone (calcined kaolin SP-33, BASF Corporation, NJ, USA), and Lactose monohydrate (Foremost Farms, WI, USA). The material used to demonstrate the predictive model was a calcined zeolite which we term “Material A” (Albermarle Inc., Amsterdam, the Netherlands). The reason for using calcined zeolite to validate the predictive model is that it is an industrially relevant cohesive material that can potentially lead to failures during process and product development. Particle size information and conditioned bulk density of all the materials is listed in Table 1. Particle size was determined using a laser-diffraction (LS-13320) analyzer with Tornado Dry Powder System (Beckmann-Coulter, Brea, California, USA). Conditioned bulk density was measured after the standard conditioning cycle of the shear cell test, which is further detailed in section 2.2. The results were for a limited number of powders with a limited range of particle sizes. The utility of this approach would be improved by adding new materials with different particle sizes to the database.

2.2 Material flow property measurements

The following tests were performed: compressibility tests, permeability tests, shear cell tests, and dynamic flow tests.

Compressibility test

The compressibility test is part of the Freeman Technology FT4 Powder Rheometer suite (Freeman Technology Inc., Worcestershire, UK). The test measured change of powder bulk density as normal consolidation stress changes [32]. The powder was first conditioned by a helical blade moving downwards and moving upwards three times with a tip speed of 60 mm/s, in order to create a uniform and reproducible packing state without changing particle properties [33]. A normal force was then slowly applied by a vented piston. The normal forces range from 0.5 to 15 kPa, holding each load for 60 seconds. The change of volume due to compression was measured, and the compressibility (CPS %) was calculated as the percent change in volume after compression:

$$\text{CPS \%} = 100 * \frac{V_c - V_p}{V_c}$$

where V_c is the bulk volume after the conditioning step and V_p is the powder volume after compression.

Permeability test

The permeability test, also achieved by the FT4 powder Rheometer, measures how well air passes through a powder bed [32, 34]. After the conditioning step mentioned previously, an upward air velocity of 2 mm/s was introduced from the bottom of the powder bed. Meanwhile, a normal stress was applied by the vented piston over a range of 0.5 to 15 kPa. At each normal force, the pressure drop (PD) across the bed was recorded.

Shear cell test

A FT4 rotational shear cell was used for the shear cell test. As usual, the testing procedure consisted of four steps: conditioning, consolidation, preshearing, and shearing [35, 36]. The conditioning step has been described previously. In consolidation, a vented piston applied the initial consolidation stress (I) to the powder. The powder was then sheared to achieve a steady-state. The shear stress and normal stress were recorded and were indicated as a preshear point. After the preshear point was achieved, the normal stress was lowered and the sample was further sheared to obtain a yield point. The paired preshear-shear procedure was repeated 5 times in total at different normal stresses to obtain a yield locus [37]. Each test generated one preshear point and five yield points. In this study, four initial consolidation stresses were used: 3kPa, 6kPa, 9kPa, and 15kPa.

For each initial consolidation stress, the shear stress was plotted in terms of the normal stress, which is also known as a τ - σ diagram. While models to take into account the shape of the yield locus have been developed [38], a commonly used approach is the Mohr-Coulomb model to generate a linearized yield locus. A schematic illustration of Mohr circle analysis for a yield locus is shown in **Figure 1**. A linear regression was fitted through the yield points to obtain a mathematical expression for the linearized yield locus. The intercept of the yield locus on the shear stress τ -axis is defined as the cohesion (τ_1), which can be interpreted as the shear stress required to deform the powder when no normal stress is applied. The slope of the linearized yield locus is $\tan(\phi)$, where (ϕ) is called the angle of internal friction (see Fig. 2), which is a measure of the ease with which the particles will slide past one another [39]. To extract other flow indices, a Mohr circle was then plotted passing through the origin and tangent to the yield locus. The intercept of this Mohr circle with the normal stress σ -axis is defined as the unconfined

yield strength (σ_c). A second Mohr circle was drawn tangent to the yield locus and passing through the preshear point (σ_p, τ_p), with its intercept on the σ -normal stress axis called the major principal stress (σ_1). The flow function coefficient (ffc), which is often correlated to the arching phenomenon in hoppers, is defined as the ratio between the major principal stress (σ_1) and the unconfined yield strength (σ_c) [40]:

$$ffc = \sigma_1 / \sigma_c \quad (1)$$

Dynamic flow test

Flow energy E is defined as the energy required to move a helical blade through a powder bed. The Freeman Technology FT4 dynamic test measures this energy as a function of time and shear rate [36]. The powder sample was loaded into the vessel and then conditioned using a helical blade in the same way as described previously. The blade, moving on the downward and upward traverse, was used for the testing cycle and the energy consumed to induce the powder to flow was measured. The energy can be different during this stage depending on material properties. It is strongly suggested that, for each material, the correlation between the consumed energy and the number of blade movements needs to be investigated before using the obtained flow parameter. Based on our prior knowledge, the energy required to induce powder flow usually becomes stable after the blade moving six times down and up for the materials in this study. Therefore, the conditioning step followed by a testing cycle was repeated seven times with measured flow energy $E_1 - E_7$. The flow energy required on the seventh downward blade pass is known as the basic flow energy (BFE). The energy consumed during the seventh upward traverse is called the specific energy (SE). The stability index, SI, reflects the change in flow energy over time:

$$SI = \frac{E_7}{E_1}$$

where E_1 is the flow energy required on the first downward blade pass, and E_7 is the flow energy required on the seventh downward blade pass. A SI value of 1 suggests that the material is stable and non-friable. If SI is larger than 1, it indicates that a material requires more energy to flow over time, possibly due to de-aeration, agglomeration, moisture uptake, or electrostatic charges. If SI is smaller than 1, it may be caused by material attrition or de-agglomeration [36].

Four additional repetitions were performed with different shear rates (i.e., different blade tip speeds) after the measuring the BFE and SI. The four repetitions were performed with a tip speed ranging from 10 to 100mm/s. The flow rate index (FRI) is the ratio between the required flow energy at 100mm/s and 10mm/s:

$$FRI = \frac{E_{11}}{E_8}$$

Where E_{11} is the basic flow energy at a blade tip speed of 100mm/s, and E_8 is the basic flow energy at 10mm/s. If materials are more sensitive to changes in flow rate, for example cohesive materials due to higher air content, the FRI values are typically higher [41].

In total, 30 different (but related) flow measurements were used as material property inputs for feeder performance prediction, including conditioned bulk density (CBD), compressibility% from the compressibility test, pressure drop at 15kPa, consolidation stress from the permeability test, cohesion, unconfined yield strength, major principal stress, angle of internal friction, flow function coefficient at 3kPa, 6kPa, 9kPa, and 15kPa from the shear cell tests, basic flow energy, stability index, flow rate index, specific energy from the dynamic flow test, and d10, d50, and

d90 from the particle size distribution. Three replications were performed for each test that was performed.

2.3 Feeder characterization

A commercially available twin screw loss-in-weight feeder K-Tron KT20 (Coperion K-Tron Pitman, Inc. Sewell, NJ, USA) was used in the study. A loss-in-weight feeder consists of three parts: volumetric feeder, weighing platform, and a gravimetric controller, as shown in **Figure 2**. The weighing platform measures the mass of the feeder and contained powder materials in the hopper. The bottom of the feeder hopper contains a horizontal agitator that helps to fill the flights of the feed screws. The gearbox controlling the screws is a type C gearbox with maximum speed of 154 RPM.

As the feeder delivers powder materials, the gravimetric controller acquires signals from the weighing platform over time, and determines the real time feed rate. The feed rate is thus controlled by adjusting the rotation speed of the screw that dispenses the material. The feeder was placed on a plane lab bench. Feeder characterization experiments were performed by using a gain-in-weight catch scale (OHAUS adventurer, OHAUS Corporation, Parsippany, NJ, USA) to record the weight of powder dispensed by the feeder every 1s for all tests. Buckets were used to collect the samples. The feeder was connected to an external laptop. Besides data from the catch scale, feeder data from the gravimetric control box was also recorded to ensure consistent control capability, including time, set point, mass flow, screw speed, drive command, and perturbation value. Three feeder screws were available in this study: fine concave screws (FCS), coarse concave screws (CCS), and fine auger screws (FAS). The experimental set-up and screws are

shown in **Figure 3**. The feeder was first calibrated with 100% fill level at volumetric mode, without engaging the feeder gravimetric control system, to determine the maximal volumetric capacity. The calibration procedure was performed three times and the average was recorded as the initial average feed factor. The averaged initial feeder factor is the control value that refers to the capacity of the feeder at 100% of the control magnitude [11].

After calibration in volumetric mode, the feeder was then run in gravimetric mode starting at the 80% fill level. The fine concave screw was firstly used because it has the smallest capacity among the three screws. For each material, the set point for the three screws tested was kept constant and set at 50% and 80% of the initial average feed factor of the fine concave screw. For each screw, the experiment was run for 20 min until the hopper reached approximately 60% fill level. The data obtained from the catch scale and the control box were used for screw comparisons. Defining the target mass feed rate as m , the actual mass feed rate, $\dot{m}_i$, at the i th time point can be calculated by:

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

Since the set point for each material may not be the same, the relative standard deviation (RSD) and the relative deviation between the set point and the mean (RDM) were used as criteria for feeder performance:

$$\sigma = \sqrt{\frac{\sum_{i=1}^n (\dot{m}_i - \bar{\dot{m}})^2}{n - 1}}$$

$$\text{RSD} = \frac{\sigma}{\bar{\dot{m}}}$$

$$\text{RDM} = \frac{|\dot{m}_i - \bar{\dot{m}}|}{\bar{\dot{m}}}$$

where σ is the standard deviation, $\bar{\dot{m}}$ is the arithmetic mean of the actual mass feed rate, and n is the number of time points. A moving average of 5 seconds of the data obtained from the catch scale was used to calculate the RSD and RDM ($\Delta t=5$) [42, 43]. RSD quantifies the consistency of the mass feed rate, and RDM suggests the difference between the actual mass feed rate and the target mass feed rate. In this study, the aim is to find the screw that provides the best feeder performance, namely performance with lowest RSD and RDM. For data analysis, experimental data for the first 5 seconds and the last 10 seconds were removed since there may be affected by significant perturbations. For the same reason, data of a period of 20 seconds were also removed if changing of the bucket was needed during the experiment [11].

2.4 Multivariate analysis

Multivariate data analysis was performed using The Unscrambler X 10.2 (Camo, Oslo, Norway). Since material properties have different units and their ranges have different scales, the data was first preprocessed using Z-score normalization. The z scores were calculated as follows:

$$z = \frac{x - \mu}{\sigma}$$

where μ is the average of each property and σ is the standard deviation from the average. Data after z-score normalization are centered around 0 with a standard deviation of 1, which is a common requirement for most machine learning estimators, especially for clustering analysis, such as PCA, when comparing similarities between samples is needed based on certain distance measures [44, 45].

Two approaches to predict feeder performance are discussed in this study: Principal component analysis followed by similarity scoring (PCA-SS), and Partial least squares regression (PLSR). Both approaches identify hidden structures in the material property data set and use a vector space transform to reduce the dimensionality of large data sets [46]. The difference between the two approaches is that PLSR directly regresses the data in reduced dimensions to feeder performance, while PCA-SS firstly identifies similarity between materials and uses feeder performance of the most similar material to predict results.

In the PCA-SS approach, a PCA model was used to represent the z-score normalized material property data set (X) in a reduced dimension (principal component space) such that the major axes of variability are identified [47]. The data set X can be decomposed, based on the equation below, into a set of scores (T) and loadings (P), while the remaining variability is modeled as random error (ϵ).

$$X = TP^T + \epsilon$$

The columns T represent principal component (PC) scores of each material in the projected space; loadings P represent the significance of each material property in each principal component. Both T and P are obtained from eigenvectors and eigenvalues of the covariance matrix of X . A good indication of similarity between two materials is a measure of the distance between them [48]. Principal components (PCs) [49] are orthogonal to each other, and each is associated with the value that explains the proportion of variability in the data set. Usually, only a few PCs are retained based on their statistical significance. The similarity scores between material a and material b can thus be calculated based on weighted Euclidean distance d_w :

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

where n is the total number of principal components selected in the model, a_i is the score of material a in the i th principal component, b_i is the score of material b in the i th principal component, w_i is the weight of the i th principal component, namely the relative variability explained by the i th principal component:

$$0 < w_i < 1$$

The similarity score is based on the concept of distance between objects, which quantifies the similarity or dissimilarity between observations in the data set [48]. A relatively small value of d_w suggests similarity between materials.

Partial least squares regression, also known as projection to latent structure regression, was used also to correlate a number of predictor variables X with response variables Y by finding the latent variables in the data set. The calculation of latent variables in the PLSR approach takes into account the response variables so that the linear combinations have maximal covariance [49]. In this study, the aim is to understand how varying flow indices impact feeder performance for different materials. Therefore, flow properties of six powder materials, covering a wide range of flow properties, were used to develop the calibration model for RSD and RDM obtained from feeder characterization experiments. Cross-validation (CV) was used for initial method evaluation. CV was performed by developing six parallel regression models from reduced data with one of the materials deleted, which is also called leave-one-out cross validation. In addition, the model was used to predict feeder RSD and RDM of one additional material, material A. A

confirmatory feeder experiment for material A was then performed using the same procedure described in section 2.3. The predicted results were compared with the experimental results.

Results and discussion

To demonstrate the suitability of the proposed approaches, Material A was used as the prediction material. This material belongs to a group of calcined zeolite materials for catalyst manufacturing processes. Flow properties of Material A were firstly characterized, and each flow parameter was compared with all other materials that were already characterized. The methodology used in this study is based on efforts to establish a material library, where commonly used powder materials are characterized by a standard set of properties [50]. The existing materials in the library serve as a calibration data set that is representative of a certain range of material properties of interest. Both approaches are, in general, data-driven, so it is important to ensure that each flow parameter of Material A is between the minimum and maximum values of existing materials [51]. **Figure 4** compares the results from shear cell tests at an initial consolidation stress of 9kPa. The major principal stress relates to a material property during steady-steady flow under a certain normal stress. Unconfined yield strength, cohesion, and angle of internal friction are known as characteristic values when a material achieves incipient flow [35]. The flow function coefficient (ffc) is often used to rank the flowability of a material, where a larger ffc value indicates a more free-flowing material. The materials can be ranked in terms of ffc from highest to lowest as follows: coarse alumina, zeolite, lactose, fine alumina, satintone, Material A, and fine zeolite. The angle of internal friction suggests frictional forces between the particles, while the cohesion reflects the cohesive forces between particles. According to a previous study, the cohesion and the flow function coefficient follow an intrinsic power correlation using the Mohr-Coulomb model [35]. Therefore, the trends shown by the

angle of internal friction and the flow function coefficient are not consistent. The results from shear cell tests have confirmed that powder flow cannot be described by a single parameter. Shear cell test results at initial consolidation stresses of 3kPa, 6kPa, and 15kPa are not shown in **Figure 4**, but it was confirmed that, for each flow index, data for Material A was between the minimum value and maximum value of six calibration materials.

Figure 5 shows results from compressibility tests and permeability tests for all materials. Typically, a lower compressibility percent corresponds to a material with lower cohesion and better flowability. Based on compressibility results, the material flowability can be ranked from highest to lowest as follows: Coarse alumina, zeolite, lactose, Material A, fine alumina, fine zeolite, and satintone. Interestingly, material flowability ranking in terms of compressibility percent was different from the one based on ffc. In other words, materials with higher ffc do not necessarily have higher values of compressibility percent. Similarly, pressure drop, obtained from permeability tests, did not have a linear correlation with compressibility percent. For example, while both fine alumina and fine zeolite had relatively high values of compressibility percent, fine alumina had much larger pressure drop than fine zeolite, possibly due to differences of pore volume inside the particles. The permeability measured the change of powder bed volume with resistance to applied normal stress when air is introduced from the bottom of the powder bed. The lack of correlation between the permeability and compressibility results is possibly due to the fact that some of the materials in this study have intra-particle pores [52].

Figure 6 includes results from the dynamic test. As mentioned previously, the basic flow energy (BFE) measures the energy required to stir a blade through a sample. Generally, if a material is free flowing, it requires low energy to move the powder, resulting in a low BFE. When a material becomes more porous, the BFE will also be low, as the blade is in contact with the low

density particle bed. Interestingly, among all the materials characterized, coarse alumina was the least compressible material and satintone was the most compressible material. Both materials had a relatively higher value of BFE compared to other materials, suggesting that BFE can be an orthogonal parameter to describe powder flow in addition to the compressibility.. All materials in this study had a stability index (SI) close to 1.0, indicating that the materials are relatively stable and non-friable. Flow rate index (FRI) measures the material sensitivity to flow rate. Most materials in this study had an FRI value of between 1.5 to 3.0, corresponding to a modest increase in flow energy at a higher flow rate. Lactose, with an FRI value of 1.0, was considered as a flow rate insensitive material.

In summary, two conclusions can be made based on the material characterization results: i) For all the flow indices considered in this study, Material A was in the range between the maximal value and minimal value of the other six materials. The behavior of Material A can thus in principle be predicted based on interpolation of the material properties. ii) Comparing materials based on a single flow index, also called a univariate approach, is not sufficient. As described earlier, each flow parameter reflects a different aspect of flow behavior. Furthermore, results from conducting multiple univariate comparisons may often be difficult to interpret. In fact, the univariate comparison approach does not take into account the correlation between different parameters. Results have shown that flow parameters do not vary independently, but the manner in which they correlate with each other is a critical material characteristic. Therefore, a multivariate analysis was performed to compare materials with different flow properties.

In the multivariate analysis, not only the isolated individual flow index, but also the relationships between different indices were included. All material property data can be considered and analyzed simultaneously, providing greater statistical power (relative to a univariate comparison

[53]). A PCA model was developed including Material A and the six calibration materials. As the aim of this study is to identify materials with similar properties, enough principal components were retained to ensure that the model captured at least 95% of the overall variability in the data set. In addition, cross-validation was used to avoid model over-fitting when most of the variability was included in the model. As shown in **Figure 7**, the first four principal components were found to explain 96.4% of the total variability in the dataset. These were also the components with the highest explained variability in validation, and for this reason, four components were retained in the model to be used in subsequent calculations. A score plot was used to visualize how each material is projected into the reduced dimension space. As shown in **Figure 8**, a cubic score plot, including the first three principal components that explained 91.4% of the variability of the data set, suggests that the six calibration materials represented a wide range of materials with varying flow properties. If materials have the same properties, they will be projected to the same location in the score plot. Materials that are projected close to each other indicate similar flow behavior. Figure 8 also suggests that Material A is projected to the center area of the cluster, suggesting that, in a multivariate perspective, the flow properties of Material A is within the range of the existing materials. When an outlier material needs to be assessed before prediction, Hotelling's T^2 test is recommended in conjunction with the score plot [54]. To quantify the distance between two materials, a weighted Euclidean distance was calculated based on the first four principal components, as described in section 2.4. The similarity score outputs are presented in **Figure 9**. The material most similar to Material A was fine zeolite. The identified similarity between Material A and fine zeolite using PCA-SS was further confirmed in the following feeder characterization experiment.

The relative standard deviation (RSD) and relative deviation from the mean (RDM) using different screws were used for comparison of the feeder performance for each calibration material, as shown in **Figure 10** and **Figure 11**, respectively. Consistent with the previous knowledge, materials with different flow properties lead to different feeding performances, and therefore different screws should be selected in order to achieve consistent and accurate delivery of materials [55]. For example, for coarse alumina, which is very free-flowing and can easily fill the flights of the screws, all the screws were found to be able to achieve stable feeding at both the predefined feed rates. The feeding performance of some less free-flowing materials, such as lactose monohydrate and zeolite, was found to be heavily dependent on the selection of the screw and feed rate. For lactose, the fine concave screw (FCS) achieved the best performance at a feed rate of 7.0 kg/hr and the coarse concave screw (CCS) became optimal when the feed rate was increased to 11.2kg/hr. For zeolite, the FCS had the lowest RSD and RDM at feed rates of 3.0 kg/hr and 4.9 kg/hr. However, use of the fine auger screw (FAS) at a feed rate of 4.9 kg/hr significantly increased the RSD and RDM. For the cohesive materials studied here, the selection of the optimal screw was consistent at the two feed rates investigated in this study. Specifically, the FAS was considered as the optimal selection for satintone at feed rates of 2.1 kg/hr and 3.4 kg/hr, and for fine alumina at 4.3 kg/hr and 6.8kg/hr. The FCS was selected for fine zeolite, both at 4.7kg/hr and at 7.5kg/hr.

As fine zeolite had the shortest weighted Euclidean distance to Material A, the FCS was predicted to be the optimal screw for Material A. To validate the prediction, a feeder experiment was performed using Material A and the results were compared to that of fine zeolite. As shown in **Figure 12**, the FCS was the optimal screw for both materials. However, it was noticed in **Figure 12(a)** that the FAS had a higher RSD than the CCS for fine zeolite, while for Material A

the order reversed. Nevertheless, the overall performance of Material A and fine zeolite was relatively similar.

Surprisingly, a strong correlation was observed between the initial feed factor and the score of the first principal component for all three screws, as shown in **Figure 13**. Since the first principal component is a linear combination of the measured flow indices, the initial feed factor can be directly predicted based on the observed correlation. The initial feed factor is an important process parameter that refers to the maximal capacity for each screw. A typical operation set point during the feeding process for each screw is usually 20-80% of its maximal capacity. Therefore, the initial feed factor is required to determine the operational space. To obtain the initial feed factor, the hopper needs to achieve a 100% fill level, which requires a comparably larger amount of materials relative to the amount required for the material property measurements. In the case presented here, 2-6 kg of each material was used, depending on the material bulk density in order to obtain the initial feed factor, while the amount of material required for characterization was 0.2-0.5kg. It is common that in early process and product development the amount of material is often limited. In this case, the predictive correlation can be used to determine the initial feed factor based on material properties as represented by the scores of the first principal component. Further examination suggests that the pressure drop from the permeability test and the stability index from the dynamic flow test had small loadings for the first principal component, while the remaining measurements were highly correlated and had relatively higher loadings. Methods will be explained in a future paper for using PCA-SS approach to reduce the number of measurements, and to understand the physical meaning of the first principal component correlated to the initial feed factor.

The workflow of PCA-SS approach is described in **Figure 14**. In general, when a new material is received, it is first characterized by the flow property measurements described in section 2.2. A PCA is performed, including the new material and all existing materials that have been previously characterized. By knowing the PC score of each material and weight of each PC, the weighted Euclidean distance can be calculated. Distances between the new material and all other materials can be ranked, and the material with the smallest distance to the new material can be identified. Based on the assumption that under the same feeder parameter settings, materials with similar flow properties have similar feeder performance, the optimal feeder screw type for the new material can be predicted based on existing knowledge of the material with the smallest weighted Euclidean distance. More importantly, the new material properties are automatically added to the material library. As the number of characterized materials expands, the accuracy of identifying similar materials will also be improved.

The advantage of the PCA-SS approach is the ability to quickly identify similar materials considering all the available flow property measurements. The predicted material is also included when developing the PCA model, which enables the model to fully explore the overall data structure and patterns. The prediction error can be reduced significantly by using similarity scores, instead of projecting the predicted material data to the existing PCA models. When the material available is limited, this approach is especially useful to identify surrogate material to quickly identify the design space and avoid failure modes during process development. Additionally, the selected surrogate material can be used to accelerate process scale-up and technology transfer. However, there are limitations to the PCA-SS approach. The approach is based on an assumption that materials with similar flow properties will require a similar screw in order to achieve optimal feeding performance. If the calibration materials do not cover a wide

range of flow properties, the difficulty in finding similar materials may potentially increase prediction error. In addition, the optimal screw was predicted based only on the material that is most similar. Future work is needed to validate the proposed method using a larger industrially relevant material database.

To consider the potential quantitative correlation between material properties and feeder performance, PLSR models can be developed. In this study, four factor non-hierarchical PLS models were fitted to the material property data consisting of six calibration materials and 30 flow parameters. The response variables were the RSD and RDM for each screw at feed rates corresponding to 80% of the initial feed factor of the FCS. The cumulative percent variance explained by the four factors in all the models was above 95%. **Figure 15** suggests that good parity was observed between the predicted and actual y-values (RSD or RDM). The root mean square error of calibration (RMSEC) and root mean square error of cross validation (RMSECV) were also shown. The regression coefficients for the RSD models are depicted in **Figure 16**. The regression coefficients indicate the contribution of each material flow property to the prediction models, with higher absolute values of the coefficients indicating larger contributions. A positive value of the coefficients indicates a positive correlation with the RSD, and a negative coefficient value indicates inverse correlation. Material flow properties with relatively small coefficients suggest that the contribution to the prediction is small. **Figure 16** suggests that material properties contribute to feeder performance differently when different screws are selected. For example, pressure drop (PD) from the permeability test had a significant effect on the RSD when using the FCS and FAS, while its contribution when using the CCS was relatively small. Also, interestingly, the PD increased the RSD when using the FCS, but decreased the RSD when using the FAS.

The RSD and RDM of a material during the feeding process can be predicted based on the obtained regression coefficients and material flow properties. **Figure 17** shows the predicted results of Material A in comparison to the measured results. It can be seen that the developed PLSR models were able to predict RSD and RDM values that are reasonably similar to the experimental results. By comparing the predicted RSD and RDM values using different screws, the FCS was predicted to be the optimal screw for Material A, which was consistent with predicted results using the PCA-SS approach, as well as the experimental results. The workflow using the PLSR approach can be summarized in **Figure 18**. When a new material is received, its properties are first characterized. The material property data is then projected to the PLSR model. The model outputs are the RSD and RDM using different screws. The optimal screw, with the lowest predicted RSD and RDM value, is then selected. Confirmatory experiments, using the screw from the predicted results, are performed. Importantly, models are consistently maintained and updated by adding the confirmatory experimental results to the model.

It is worth noting that characterization techniques used in this study can potentially save material and time compared to feeder tests. The amount of time required for a particle size test, a compressibility test, a permeability test, a shear cell test, and a dynamic flow test can be as small as approximately 3min, 10min, 10min, 15min and 15min, respectively. For a typical material with bulk density of 0.6 g/ml, the amount required to perform a test is 5g, 6g, 6g, 6g and 15g for the particle size test, the compressibility test, the permeability test, the shear cell test, and the dynamic flow test, respectively. With three replications, the total amount of material used is approximately 100g (for all the characterization tests we carried out) and properties can be collected in about four hours. This assumes that one does not reuse any of the materials for any of the tests. If it is possible to reuse material for the tests, then even less material can be used.

The amount of materials used is significantly smaller than that consumed in the feeder experiments. In the feeder experiments performed in this study (which required about 30min), at least 1.5kg of material would be consumed at a feed rate of 3kg/hr for one type of screw, and 5kg would be consumed at a feed rate of 10kg/hr. If we consider running experiments for three different screws at (typical feed rates for this study of) 3kg/hr, 5kg/hr, 7kg/hr, and 10kg/hr, then we would consume approximately 12.5kg of material (assuming we do not reuse any of the material). If we consider feeders with larger feed rates, then this methodology would save even more materials. For one material and one feeder set point, using one screw, our feeder experiment took about 30min. However, if you want to characterize more than eight conditions, then you will start to save time. e.g. four different screws at four different flow rates would require approximately eight hours, which is twice the time needed for the characterization experiments. As one moves to manufacturing scale with an even higher throughput, the methodology provides an efficient approach to significantly lower the development costs. The proposed method is a proof-of-concept of using data-driven models to accelerate process design and lower development costs. The established database containing material properties can be consistently expanded and can potentially be used to correlate with the performance of other processes.

While multivariate analysis has been used to develop predictive process performance based on material properties, it is important to note that the proposed methodology does not conflict with standard design of experiment techniques. In fact, both are statistical tools that can be used in combination to identify a design space during process and product development [56]. For the multivariate approach, the size of the data set is important to both the approaches discussed in this study. For the PCA-SS approach, as more materials are added, model knowledge to predict

feeder performance is consistently expanded and accumulated, increasing the likelihood of finding similar materials for a given new material. For the PLSR approach, models are maintained and updated when feeder performance of more materials are obtained during process development, continuously improving the prediction accuracy for future materials. Further work is required to test if the proposed approaches can be validated across a wider range of material flow properties that were not considered in this study, for example electrostatics [57, 58], surface energy, etc. Additionally, as more flow property measurements are considered, it would be useful to combine multivariate analysis and first principal models to meaningfully select the measurements that have higher weights in terms of classification of the materials.

Conclusions

This paper examines two methodologies, PCA and PLS, to develop predictive correlations between material flow properties and feeder performance. The methods include characterization of material flow properties, characterization of feeder performance, and predictive multivariate analysis. Six calibration materials with varying flow properties were firstly characterized by five techniques. The flow properties of each material were represented by 30 flow indices. The calibration materials were characterized by feeder performance. Multivariate analysis was used to find the correlation between material properties and feeding performance. Specifically, two approaches were discussed and compared. The PCA-SS approach is based on weighted Euclidean distance, calculated after performing the principal component analysis on the entire data set. The material that has the shortest distance to a given new material can be considered to have similar flow behavior. The PLSR approach further quantifies the correlation between material properties, projected into a reduced dimensional space, and feeder performance, represented by the RSD and RDM.

This study shows that material flow properties affect feeder performance, and therefore the screw that achieves the best feeding performance is heavily dependent on the material flow properties. Experimental results in combination with multivariate analysis suggest that material flow properties and feeding performance are highly correlated. Both approaches discussed in this study were confirmed by cross-validation and prediction of one additional material. The predicted feeding results were, in general, in good agreement with the experimental results. The work presented here has shown an efficient approach to correlate material properties with process performance using multivariate analysis. This approach is especially powerful when the amount of a given new material is limited, or if the new material is expensive or dangerous. The data-driven models can significantly reduce the amount of materials and time needed for process development thus lowering development costs.

The initial feed factor and scores of the first principal component for each material were found to be strongly correlated. By characterizing flow properties, the initial feed factor, or maximal capacity, of each screw can be predicted, which enables a quick identification of operational ranges. By using the PCA-SS method, a placebo material with similar flow behavior can be identified. When a material is not available in large amounts, placebo materials can be used for process development. Additionally, instead of selecting optimal tooling based on empirical knowledge and a trial-and-error method, feeding performance using different screws can be directly quantified, predicted and compared by developing predictive PLSR models.

Importantly, for any data-driven models, the size of the data set is important for prediction. Therefore, the methodology should be implemented as a self-updating knowledge reservoir. The more materials and properties that the data base includes, the better will be the predictive power of the models. It is also important to notice that the same type of models can be augmented by

additional measurements performed during process scale-up, validation, and commercial manufacturing in order to capture process knowledge and increase predictability. At the same time, this study has been for a limited number of materials and for one feeder. Future work should examine additional materials and other feeders in order to examine if the results observed in this work carry over to other types of feeders. Future work should also test the validity of the proposed methods across a wider range of material properties, characterized by additional techniques. As more characterization techniques become available, PCA-SS can also be used to reduce the number of measurements, which will be addressed in a future publication.

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Captions

Table 1 Particle size and bulk density information for the materials used in the study.

Figure 1 Mohr circle analysis for shear cell tests.

Figure 2 A schematic illustration of a loss-in-weight feeder. (picture source: W. Engisch, F. Muzzio[11])

Figure 3 (a) Experimental set-up for characterization of feeding performance for each material. Available screws in this study are (b) fine concave screw, (c) fine auger screw, and (d) coarse concave screw.

Figure 4 Material characterization results from shear cell tests at an initial consolidation stress of 9 kPa.

Figure 5 Material characterization results from compressibility tests and permeability tests.

Figure 6 Material characterization results from dynamic flow tests.

Figure 7 The first four principal components were selected for the PCA model.

Figure 8 A cubic score plot was used to visualize how different materials are distributed in the projected spaces. The coordinates of each material are shown as the scores of each principal component. The similarity score based on weighted Euclidean distance can be calculated to further quantify similarity or dissimilarity between different materials.

Figure 9 Weighted Euclidean distance between Material A and other existing materials were calculated. Based on the concept of distance between objects in the reduced dimensions, fine zeolite was identified to be the most similar material to Material A. In other words, the feeder performance of fine zeolite can be used to help the prediction of Material A.

Figure 10 The gravimetric feeding performance for six calibration materials were characterized. The feeding performance was represented by the relative standard deviation (RSD), for each material using three available screws.

Figure 11 The gravimetric feeding performance for six calibration materials were characterized. The feeding performance was represented by the relative deviation from the mean (RDM), for each material using three available screws.

Figure 12 Comparison of the feeding performance between fine zeolite and Material A at (a) 50% of the initial feed factor for the fine concave screw and at (b) 80% of the initial feed factor for the fine concave screw. Screw type 1 corresponds to the fine concave screw, screw type 2 corresponds to the fine auger screw, and screw type 3 corresponds to the coarse concave screw.

Figure 13 (a) Initial feed factor during volumetric calibration for each material using the three available screws. (b) A strong relation between the obtained initial feed factors and the scores of the first principal component.

Figure 14 Workflow of the principal component analysis - similarity score (PCA-SS) approach.

Figure 15 Predicted versus reference parity plot for feeding performance, represented by RSD and RDM, for three screws.

Figure 16 Regression coefficients for PLSR models of RSD prediction.

Figure 17 Predicted feeding results for Material A, in comparison with experimental results. Screw type 1 corresponds to the fine concave screw, screw type 2 corresponds to the fine auger screw, and screw type 3 corresponds to the coarse concave screw.

Figure 18 Workflow of the partial least squares regression (PLSR) approach to predict feeder performance based on material flow properties.

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Figure 1

Figure 2

Figure 3_revised

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11

Figure 12

Figure 13

(a)

(b)

Figure 14

Figure 15

Figure 16

Figure 17

Figure 18

Table 1 Particle size and bulk density information for the materials used in the study.

Material	d10 (μm)	d50 (μm)	d90 (μm)	Conditioned bulk density (g/ml)
Coarse γ -alumina	11.16	59.37	122.19	0.97
Fine γ -alumina	0.99	4.07	11.46	0.31
Fine zeolite	0.94	3.85	5.81	0.86
Zeolite Y-type CBV100	0.77	2.99	10.44	0.25
Satintone (calcined kaolin SP-33)	0.92	6.09	21.14	0.52
Lactose monohydrate	10.32	63.54	157.78	0.66
Material A	0.80	2.33	7.59	0.31