Outline/Draft for IFPRI Systems Engineering Framework

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This draft summarizes content for the IFPRI Systems Engineering Framework. It builds on the previous Forward Framework document that was included in the 2018 AGM materials[1] and proposes additional review scope relevant to the initial brief from 2016, providing perspective on Systems Engineering (SE) as it applies to particulate processing. It includes open-ended questions (*blue italics*) intended to seed further discussion within IFPRI and the broader community. Feedback on these and other questions is welcome and can be addressed in the full review, teleconferences, and/or AGM programming discussions.

An intent of the original 2016 brief was to identify topics for an IFPRI workshop having a Systems Engineering focus. At the 2018 AGM, we planned to move forward opportunistically with a Granulation Systems Engineering Workshop, to be held at PARTEC in Nuremberg, 12-April-2019, organized jointly by the International Fine Particle Research Institute (IFPRI) and the Working Party on Agglomeration of European Federation of Chemical Engineering (EFCE).[*] My original plan for this review was to focus on granulation as an example for the Systems Engineering Framework, including the workshop and its proceedings as content for the review. The exercise of writing the review would also meet the need of documenting the workshop. However, joint workshop planning efforts have not progressed, forcing us to cancel the workshop. An alternate review plan is not proposed (Table 1) with detail following.

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[*] The proposal for the Granulation Systems Engineering Workshop, joint with IFPRI and EFCE, was based on ad-hoc discussions including Stefan Heinrich, Satoru Watano, Willie Hendrickson and Paul Mort at the April 2018 WCPT. At that meeting, we agreed on content including:

- Workshop Announcement: Systems Engineering Framework for Granulation.
- Workshop Objective: The objective of the workshop is to advance a Systems Engineering framework for a Granulation Processes and associated Products. The focus includes process and product models, process sensors, and methods needed to support better integration of Performance-Structure-Process relations. We will further consider advances in unit operations and supporting physical technologies needed to achieve said objective. Organization of the Workshop is a joint effort by the International Fine Particle Research Institute (IFPRI) and the Working Party on Agglomeration of European Federation of Chemical Engineering (EFCE).
- The Workshop will be held on Friday 12-April-2019, as a 1-day supplement to the PARTEC meeting in Nuremberg. We will start with a Welcome Event on the evening of the 11th after the close of PARTEC.

IFPRI proposed to develop a more detailed agenda, as follows; however, we have not been able to align this with Stefan Heinrich in time for PARTEC. It is included here as a reference point.

- Workshop Agenda: The Workshop is designed as an interactive event aimed at describing the pre-competitive scope of Systems Engineering that is relevant and required to advance both academic research and industrial practice of granulation. A sequence of three sessions with address:
  - Systems framework for product design using granules as a product form or as an intermediate to a product, with a focus on product efficacy;
  - Systems approach to process design having integrated unit operations, focus on process efficiency and control;
  - Critical needs for modeling and measurement technologies, hard and soft-system approaches.

  Each session will start with a concise problem statement featuring an industrial and academic viewpoint, followed by discussion in smaller breakout groups (~6-10 people / group), then reconvening as a full group with topline sharing from the breakouts. A final session will attempt the synthesize the input of the above, and to develop an outline for publication of the outcomes.
- Workshop Pre-Work: Participants will be provided background reading in advance of the event covering:
  - Perspective on Granulation research supported IFPRI and EFCE Working Party on Agglomeration;
  - A curated list of talks scheduled for the main PARTEC meeting that are of relevance to the Workshop and their abstracts.

Proposed Review: IFPRI Systems Engineering Framework for Particulate Products and Processes

Table 1. Summary of proposed review

<u>The original brief requested:</u>

- Flowsheets used to integrate unit operations into systems, having mass and energy balances with distributed streams (particulate attributes) that are necessary to control desired product quality attributes.
- Mechanistic models of sensors (e.g. to account for systematic errors in PSD measurements of non-spherical particles by laser diffraction) that will allow better validation of models that are subsequently used for optimal design and operation.
- Additional models such as advanced process control (APC) models suitable to link many-to-many relations among process sensors, actuators, and desired product attributes.
- Sensor and measurement technologies for in-line, on-line, at-line, and inferential sensing of process streams, including distributed attributes.
- Opportunities to adapt sensor models for more efficient process control purposes.

<u>The proposed review covers:</u>

- Brief survey of flowsheet tools either in development (including academia) or commercially available. Comparative testing and/or assessment of built-in process models is not in scope. See "Process Flowsheets"
- Sensor model focusing on 2D image analysis, where 3D particles are reduced to a 2D orthographic projection based on their orientation in the field of view. See "Sensor Models." In scope:
    - Sampling: dynamic and static sampling with both front and/or back illumination.
    - Various distribution bases (number, length, area, calculated volume).
    - Length measurements (minor, major, average chord, minimum chord through centroid, maximum chord, equivalent circular diameter).
    - Shape analysis (aspect ratio, circularity, perimeter measurements).
    - Multi-modal analyses of distributed data.
- While the physics of sensors for fine powder and/or colloidal dispersions are not in scope, the use of their data for multi-modal size analysis can be included.
- Product-process relations will be given a brief overview. If a deeper dive is desired, it is necessary to narrow the scope; see discussion under "Product/Process Framework".
- For the purpose of process control, the review will consider how to combine multi-modal analyses of image data with mechanistic process models.

Product/Process Framework

System Engineering approaches have been applied across a range of petrochemical and other commodity chemical industries, balancing optimization of product quality and process efficiency in both R&D and manufacturing. In the solids processing industries, process-structure-performance relations are more complex. Some success has been achieved in application of PAT tools in the pharmaceutical industry, for example in control of liquid-solid reactors such as crystallization.[2] Zoltan Nagy's IFPRI project has furthered this capability by integrated milling of seeds in a closed loop.[3] A similar concept is explored in a seeded granulation loop, using a mill to control seed size.[4]

A broader view considers a range of product performance criteria, where products may comprise particulates and/or where particulates are a critical part of an intermediate process. In either case, the product quality depends on the combination of material input streams, particulate processes involving those streams, and subsequent assembly processes required to convert particulates into a product form.

This chain of dependency, from constituent materials, particulate processing, product design and in-use performance is illustrated by reading Figure 1 from right to left.

A Systems Engineering approach can consider the inverse problem, starting with product performance criteria, then identifying product form options, process routes, and material requirements. The systems approach enables model-based optimization of said options, providing a left-to-right perspective of the relationships in Figure 1. While IFPRI does not consider proprietary scope or claims related to product design, we can consider the SE relations in terms of derivative properties that are relevant to in-use performance.

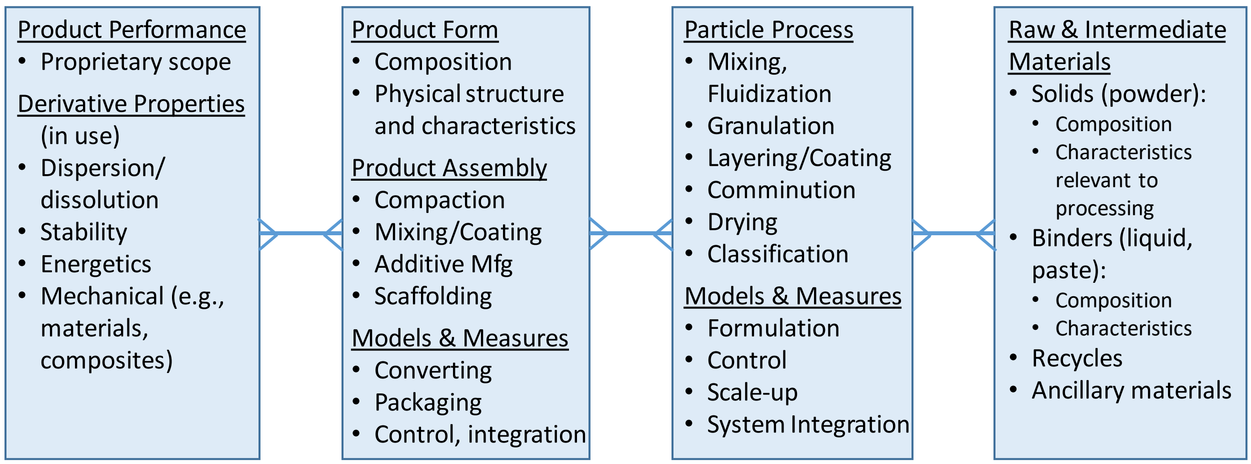

Figure 1. Relationship diagram for Process / Structure / Performance relations. Connection symbols denote many-to-many relations among blocks in the diagram:

- Input raw materials, their specification and inherent variability;
- Processing, understanding of process capability, control and adaptability to variations in raw materials, environmental conditions, etc.;
- Structure and product characteristics as an output of processing, note specifications may have distributed attributes (e.g., particle size, shape, composition in mixtures…); and
- Derivative properties and in-use performance of products as a function of composition and structure.

In my opinion, the systematic consideration of process-structure-performance relations is a key opportunity for IFPRI to exploit in the Systems Engineering area. The following three questions were a part of the Forward Framework[1] and are relevant to the discussion around Figure 1.

A potential goal for the IFPRI SE effort could be to develop and/or improve tools for process/structure/performance integration, especially in ways that facilitate inverse solutions (i.e., specifying structure and process models based on performance requirements).

Do we have sufficient models to solve the inverse problems, i.e., for a desired product performance, what must be true of product structure, processing thereof, and materials used in that process? If so, are such solutions amenable to optimization, even multi-objective optimization?

Another goal can be to describe, e.g., via expert systems, details of the many-to-many relations between blocks in Figure 2. For example, detailed mapping of the relations between processes, materials and achievable structures or specifications.

While these are important questions, and I encourage IFPRI members to push forward on their discussion and resolution, my current plan is <u>not</u> to pursue these topics in detail as a part of the full review. The overall scope is very broad; my intent is to do only a broad survey of available tools and modeling approaches, where they have been applied, and the prognosis for further application in solids processing. If there is strong interest among IFPRI companies, with specific feedback, I can add some additional perspective on specific focus areas such as:

- Granulated products requiring storage stability and in-use dispersion (e.g., detergents, fertilizers, processed foods/dietary supplements),
- Compacted particulates (e.g., pharmaceutical tablets, powder metallurgy and/or ceramics),
- Particulate composites (e.g., particles as filler in a matrix phase, or perhaps particles in a scaffolded structure).

Process Flowsheets

The following discussion is updated from the Forward Framework;[1] it asks whether current flowsheets that work by linking unit-op models are sufficient to capture the degradation of product quality and loss in operating efficiency associated with intermediate solids handling. Conversely, there are implied opportunities to improve quality and efficiency via systems approaches aimed at minimizing intermediate handling.

Industrial systems typically include primary unit operations having control objectives (e.g., crystallization, granulation, milling) along with ancillary processes (separation, classification, recycling) that can be used to further refine output quality. System models use flowsheets to integrate unit operations with flow streams having distributed characteristics. Flowsheet platforms having solids processing capability include gPROMS Formulated Products, Process Systems Enterprise (https://www.psenterprise.com/products/gproms/formulatedproducts/solids) and Dyssol under development at TUHH with funding from the DFG Priority Program SPP 1679 "Dynamic Simulation of Interconnected Solids Processes," (https://www.tuhh.de/spe/research/research-areas/flowsheet-simulation-of-solids-processes.html). Both are dynamic simulations capable of tracking distributed particle and other critical attributes, assuming suitable models are available to inform the simulations. This requires a practical combination of (1) unit-op models having distributed input and output streams with sufficient detail to predict product quality objectives; and (2) sufficient measurement data to compare against the models' predicted stream distributions. On one hand, detailed models (e.g., multi-dimensional PBM's) may be challenging to implement because they require more detailed data for comparison purposes; on the other hand, grossly simplified models may not be able to adequately predict distributed characteristics relevant to product quality specifications.

Flowsheets can be used to track process efficacy, i.e., capability to meet specifications on distributed characteristics, for example particle size, shape, composition, porosity or other structural attributes of particles. A process systems approach can be used to achieve quality objectives for distributed characteristics based on integrated monitoring and control.

Issues with process systems startup, operating efficiency, reliability and throughput capability are often traced to problems with solids handling. This is a common experience among many IFPRI members and among the broader solids-processing community. Merrow's reports from 30 years ago cited insufficient physical understanding of solids handling, raw material feeds, and solid waste-stream handing as primary contributors to poor start-up and performance of solids-processing plants.[5] Specifically, Merrow cited common problems resulting from the physical tendency of solids to "plug, stick, flow unevenly, and go where they should not (often, in the form of dust)."[6] Pinch points were cited in feed systems, classification screens, conveyors, and bucket elevators. While IFPRI has funded considerable research in dry powder flow over the interim, these physical problems persist. In fact, challenges with solids handling, transport, and ability to control flow continue to be bottlenecks to improved process systems efficiency and efficacy (product quality).

Related to the above, many particulate process systems rely on recycle streams to achieve required product specifications while minimizing material losses. Physical handling of recycles (typically particles that are either too fine and dusty or too coarse and sticky) can be challenging. A recycle strategy that minimizes handling may seem like a good idea; however, some processes such as granulation have an undesired feedback response to direct feedback of recycles (e.g., absent other control action, increasing feedback of fines in a granulation circuit will typically drive the process to produce even more fines). In such cases, surge capacity and controlled metering of recycle streams may be needed for process control. The addition of recycle surge bins and feeders pushes against challenges with solids handling.

From a product quality and quality specification perspective, solids handling and transport can be consequential, especially in cases where quality specifications rely on distributed attributes in a mixture of particles. A product that may conform to product specifications at one sampling point may degrade simply as a consequence of de-mixing and segregation during subsequent handling, e.g., by emptying storage bins, processing through packing lines, etc. More generally, particulate products are susceptible to attrition, caking and other forms of degradation that may occur in handling and storage.

On one level, systems thinking tends to separate transformations into distinct sub-systems as a means to simplify control and optimization – i.e., a simple one-to-one relation between transformations and sub-systems may be desirable in theory. In practice, such simplified approaches often ignore the consequences of handling solid materials between sub-systems.

A "solids-handling-woke" version of systems thinking seeks to minimize consequences associated with intermediate transport and handling. Combining this awareness with flowsheet modeling, one can analyze overall system efficiency and efficacy (product quality) as a function of sub-systems choices (e.g., unit operation capability) and integration thereof.

Are consequential transport and handling models sufficiently developed, i.e., relative to process efficiency, reliability, etc.? Are such models sufficient for use in flowsheets?

Recognizing the cost, operational and quality implications of solids handling, how can advances in unit operations and close-coupled integration thereof be especially advantageous to solids-processing industries?

In an effort to minimize negative consequences associated with handling and transport of intermediates and recycles, what are the priorities for advancing unit operations with control capability for multiple transformations? How can measurement and modeling of internal (unit-op) solids flow and stress fields promote and enable such developments?

Sensor Models

The review will focus on the multi-modal analysis of particle data, preferably acquired by 2D Imaging, including size, shape and other illumination effects (e.g., color, edge clarity, etc.). In principle, the multi-modal methodologies are applicable to any sensing technology that can provide cumulative distribution data. The distribution models rely on cumulative functions that can be linearized by transforming measured cumulative distributions to corresponding probability distributions. Multiple modes can be seeded according to residuals the linear regression of particle data against the probability distribution. Because of the transformation to and from cumulative distribution and probability scales, weighted regressions must be used. The review will focus on Gaussian and Stretched-Exponential distribution functions, applied on a geometric basis (i.e., Log-Normal and Weibull distributions).[7] Details of the methodology will be shared in a separate paper that is currently under preparation by the author.

The first example (Figures 2-8) uses image data from granules in a previous granular-flow study including flow field tracers.[8] The image analysis data were collected using dynamic sampling and front lighting to measure the tracers by color, where tracer granules were coated with a thin red layer (Solid Sizer, JM Canty, Buffalo, NY). This imaging method has been used for in-line process measurements. Figure 9 uses the same imaging method to explore distributions with fine and coarse tails. Figures 10-12 explore a finer powder sample using back-lit static imaging at higher magnification (Morphologi, Malvern Panalytical, Egham, UK); this is an off-line method.

Calculation of best-fit estimates of the geometric mean size, d_g, and geometric standard deviation, σ_g, can be done by weighted linear regression of x (log(d)) on y (linear model based on cumulative data) (Figure 2a). The weight functions are derived from the cumulative data (2b). In this case, the linear model is a Gaussian cumulative probability function and the cumulative distribution is based on particle 2D area data. For a single mode, log(d_g) is the x-intercept of the regression and log(σ_g) is the slope dx/dy. The multi-mode fit is derived based on the residual analysis of the single-mode linear regression (Figure 3), providing a significant reduction in the weighted mean square error, *wmse*. For display, the multi-mode results are replotted with cumulative and frequency ordinates in Figure 4.

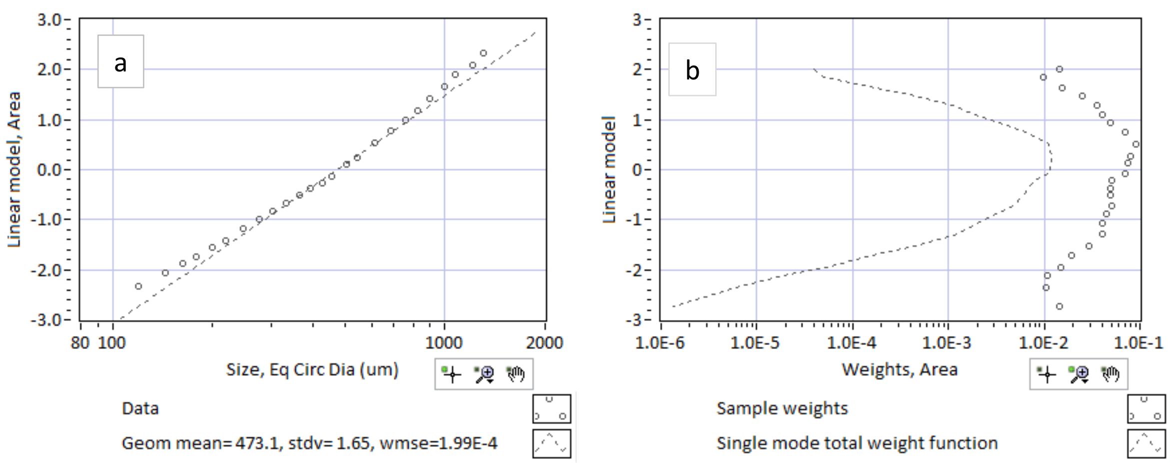

Figure 2. a) Single-mode fitting via weighted regression of log(d) on the cumulative probability function (i.e., linear model); b) weight function based on cumulative data and transformation functions.

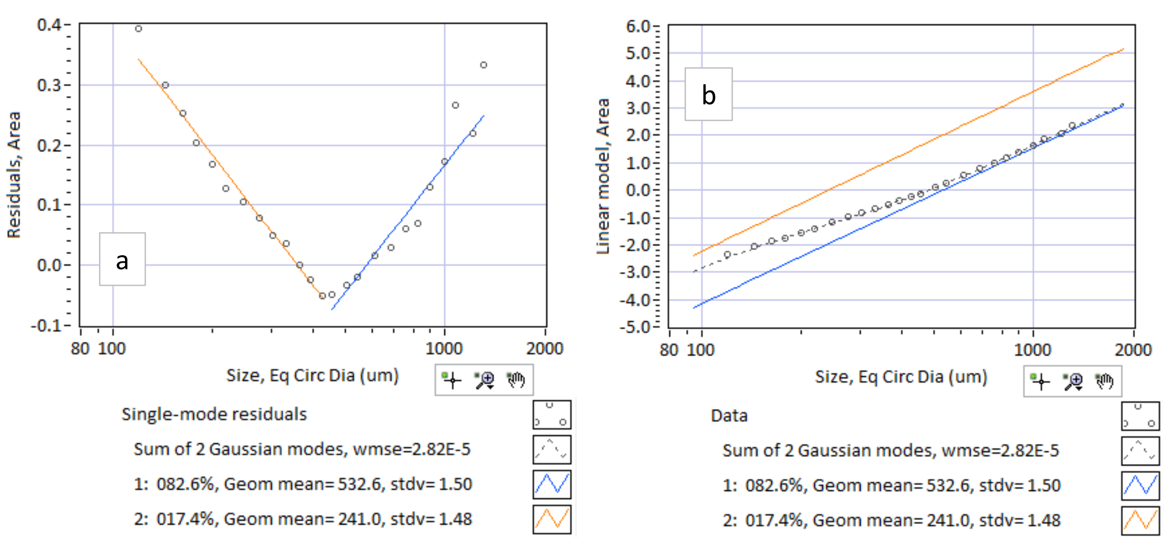

Figure 3. a) Multi-mode peak identification using residual analysis and b) resultant linear model fit.

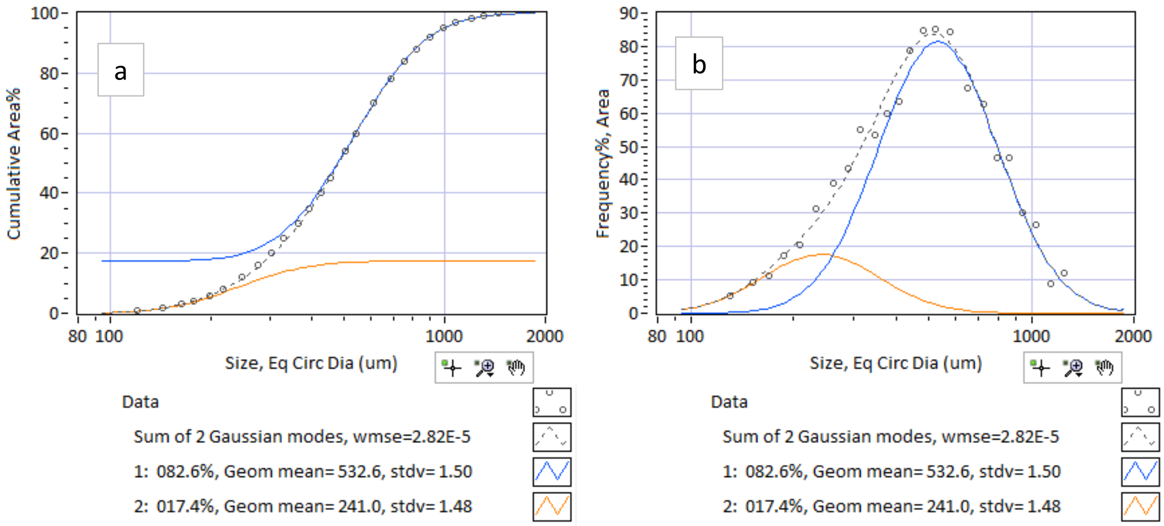

Figure 4. Replotting the multimodal fit derived above in terms of a) cumulative and b) frequency destributions. Note the frequency plot is the deriviative of the cumulative function with respect to x, where x = log(d).

Various bases may be used for the cumulative distribution including number, length (or perimeter), area, and calculated volume (Figure 5).

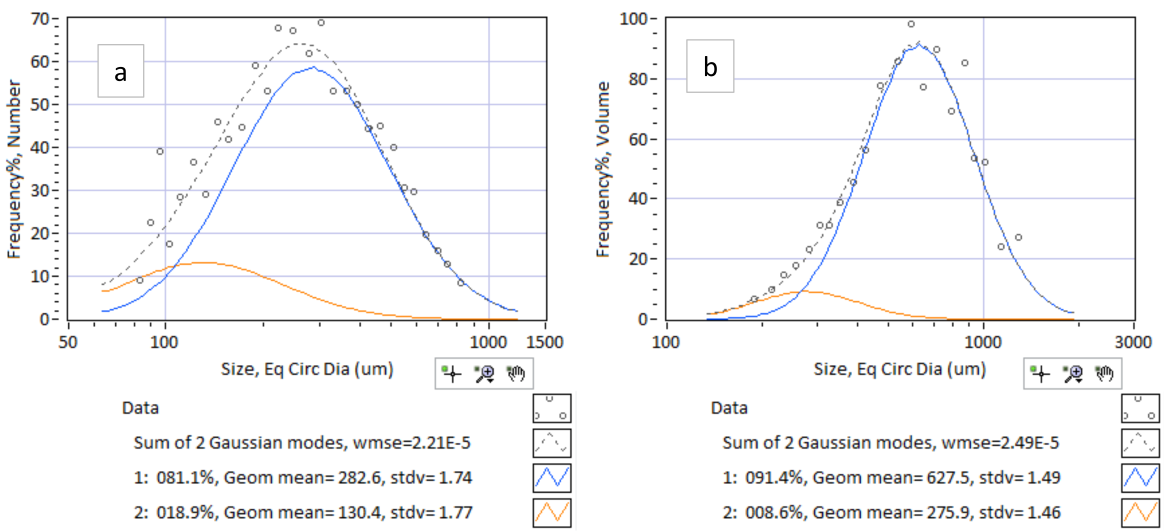

Figure 5. Comparison of bimodal fitting with a) number and b) volume-basis cumulative data; compare to 4b for area basis. In this example, bimodal character is preserved across bases; note the increase in the breadth of the number-basis distribution occurs for both modes; this behavior may vary from sample to sample.

Various length measurements may be used to describe the particles. The equivalent circular diameter shown in previous figures is calculated based on measured area of the particle. Figure 6 shows the minor and major axis length distributions; compare to Figure 4a.

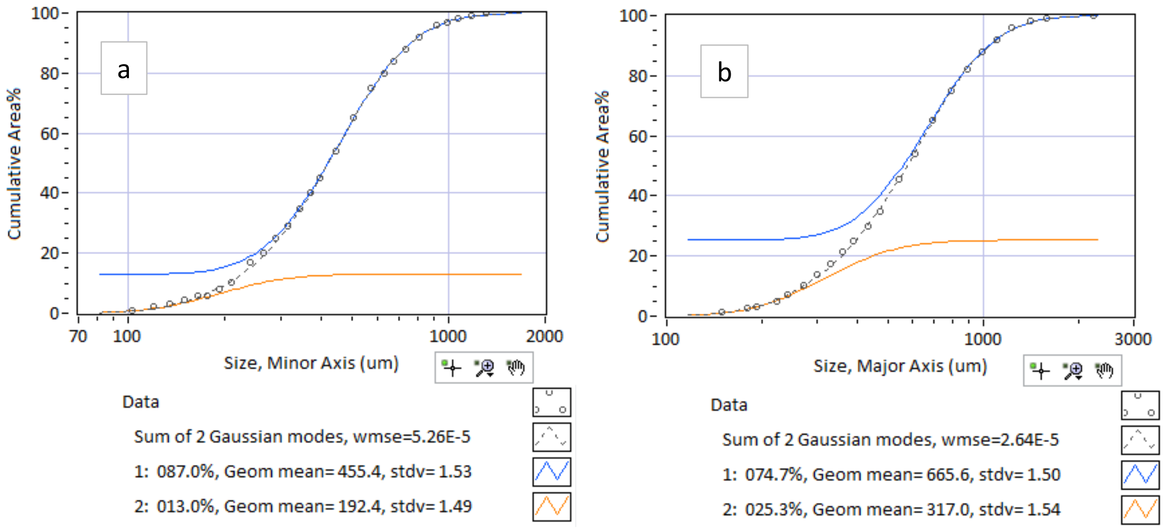

Figure 6. Comparison of bimodal fitting with a) minor axis and b) major-axis particle features; compare to 4a for equivalent circular diameter. Note the relative shift in modes as d increases.

Particle shape is often important to processing and product quality, yet it is often underspecified and/or under-controlled because of challenges in its measurement and analysis of its distribution. Figure 7 explores possible approaches to shape factor analysis and the distribution thereof. The objective is to define a reasonable descriptor of shape that is apparent from 2D imaging and is further consistent with available distribution functions.

- The Aspect Ratio (AR) is a commonly used shape factor (major/minor axis). It is observed that the log of its inverse fits reasonably well to a stretched-exponential cumulative function, where log(d*) is the intercept and n is the slope of the weighted regression. The Perimeter Ratio (PR) is defined as (measured 2D perimeter) / (equivalent elliptical perimeter of a particle having the measured area and aspect ratio). While PR has the desired feature of being a shape descriptor that is orthogonal to AR, it is not scaled to the same magnitude; i.e., the comparative use of AR and PR for shape analysis and control may require some re-scaling of PR. Both AR and PR have logical minimum values of 1. The Shape Vector (SV) is defined as the vector sum of the excess AR and PR measures: $SV = 1 + \sqrt{(AR-1)^2 + (PR-1)^2}$; its inverse is also plotted in Figure 7a.
- The circularity ratio (measured 2D perimeter) / (equivalent circular perimeter of a particle having the measured area) is another commonly used shape factor, also having a logical minimum of 1. While it is somewhat similar to PR, but is not orthogonal to AR. It is shown in 7b with a bimodal arithmetic analysis, i.e., sum of Normal distributions.

The tracer study is illustrated in Figure 8, showing multi-modal versus thresholding analyses to discern tracer particles (coated red) from the bulk of the granular distribution. In this example, the color of the base particles was white. A white particle has equal RGB color components, i.e., R = 33.3%. The bimodal analysis (8a) suggests the main mode is 33.6% Red, with a second mode having a mean of 36% red; the tracers are affecting the second mode. In comparison, 8b uses color thresholding to split the particles into two discrete populations. Using Red%=36 as a threshold, we see that the tracer population has a mean color of 40% Red and represents about 5.0% of the overall distribution.

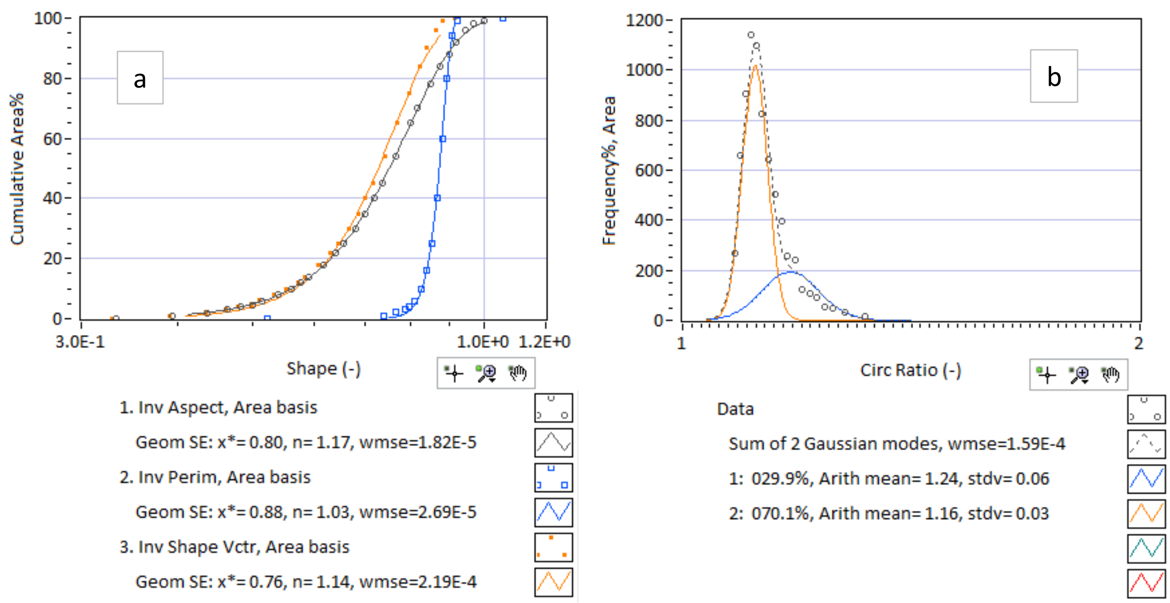

Figure 7. Shape factor distributions: a) single-mode fitting of inverse aspect ratio, inverse perimeter ratio and inverse shape vector obtained by weighted regression of the log(shape) on the stretched-exponential cumulative function. Note the aspect ratio is the dominant factor contrbuting to the shape vector; re-scaling of the perimeter ratio may be appropriate. Also note that difficutlies in accurate estimates of the perimeter can result in values of $PR < 1$; this is due to difficulty in converting from pixelated image analysis data, a sensor issue.

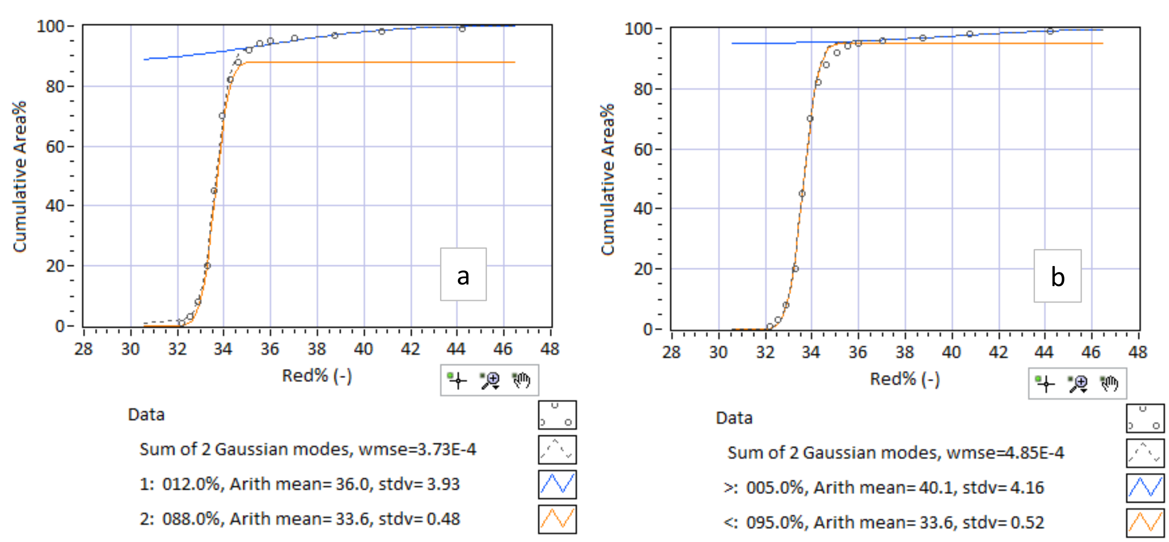

Figure 8. Particle color analysis: a) multimodal analysis using the methodology discussed herein; and b) threshold analysis to split the distribution by an independent feature, in this case, particle color.

A different granular sample is used to illustrate the use of multi-modal fitting for analysis of tails in a distribution. It is often observed that small tail fractions, either fine or coarse, can have significant impacts on processing and product quality. Yet these tails are not always evident in sensor analysis or in models. In the example, the overall distribution is well represented by a single-mode log-normal fit; in fact, the fit looks very good when viewed on a cumulative or even frequency plot (9 a, b). On the other hand, tails are evident in the linearized cumulative probability scale (9c). The use of multimode fitting (9d) elucidates small tails on both the fine (1.6%) and coarse side (1.3%) of the distribution.

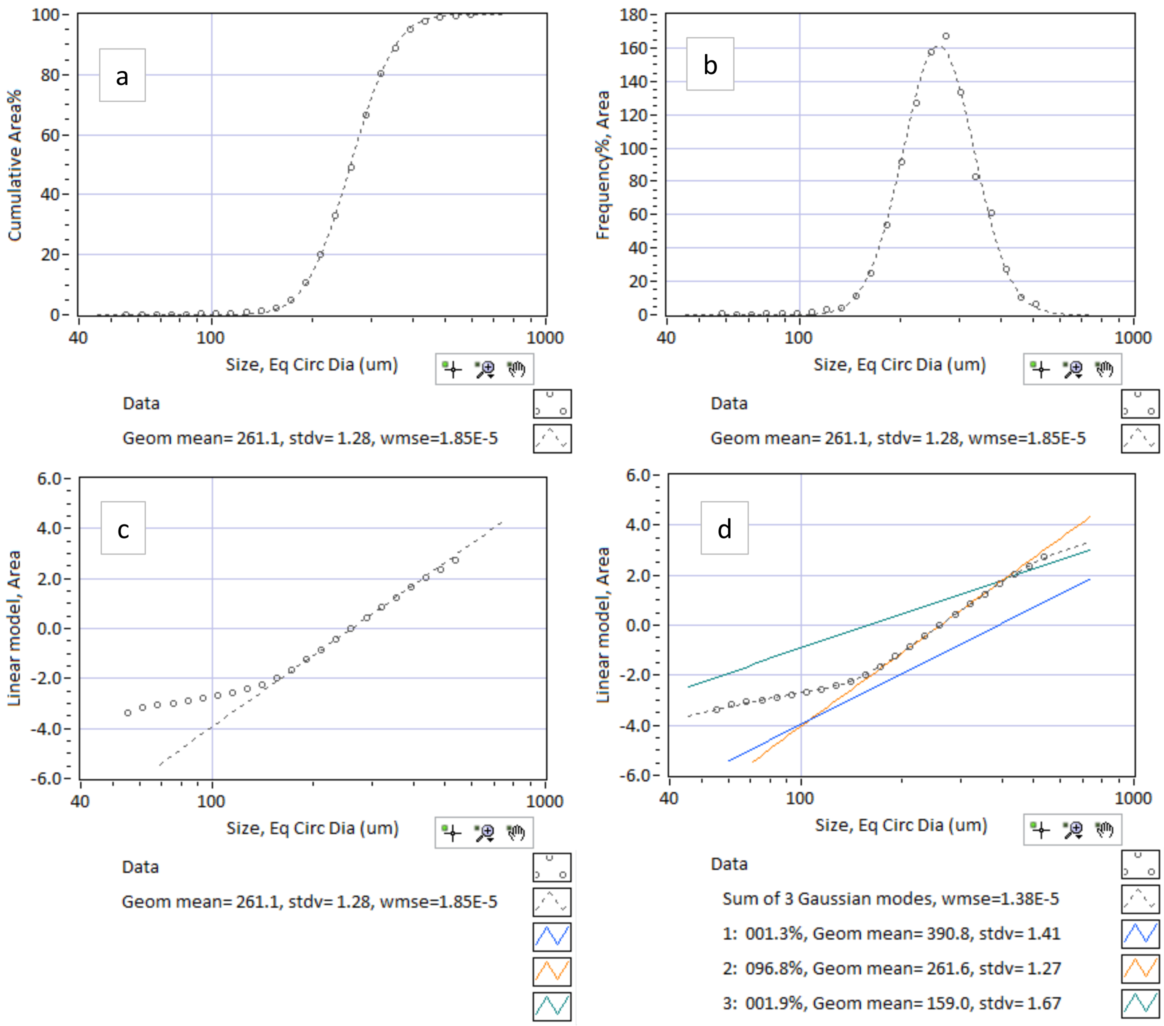

Figure 9. Multi-mode analysis of a granular sample having a distribution with minor minor fine and coarse tails. The single mode distribution look quite good when viewed on cumulative and frequency plots (a, b). The fine tail is more evident from the probability plot (c). Multimode analysis using the resuduals of the single mode regression reveal minor modes on each tail (d).

The final example is based on static sample imaging with back-lit illumination, i.e., dark particles on a light background. The sample data contains 20,000 particles from a milk powder with a substantial fraction of fines.[9] The residual analysis reveals three significant modes (Figure 10). While the shape factor analysis (Figure 11) shows reasonably good geometric fit of the inverse shape factors to the cumulative stretched-exponential distribution model, we observe a small portion of the Inverse PR distribution having illogical values, $1/PR > 1.0$. Diving deeper (Figure 12), we see there is an apparent correlation between the particle size and the illumination characteristics of the sensor, i.e., larger particles appear darker. Figure 12 is a scatterplot with each point representing a particle. The blue points have a logical $PR >= 1$; the orange points are illogical ($PR < 1$). It appears that for a given illumination characteristic, smaller particles are more likely to be under-biased in their perimeter measurement. Bottom line, for shape analysis, there appears to be a need to re-bias perimeter measurements and re-scale their derivative shape factors.

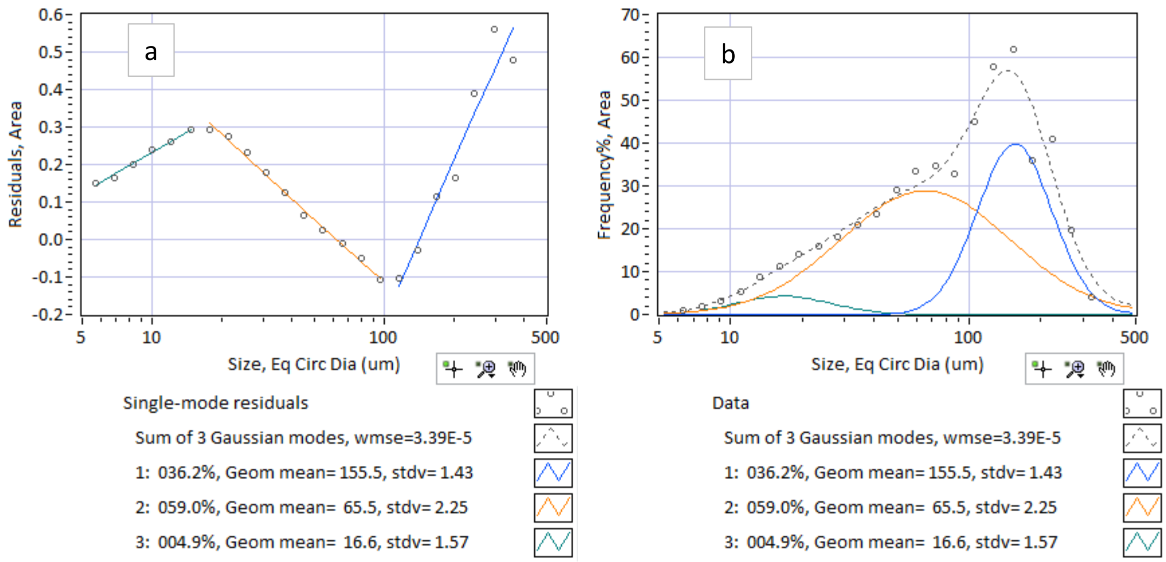

Figure 10. Multi-mode analysis of a milk powder sample. Residual analysis (a) indicates three significant modes, albeit overlapping (b).

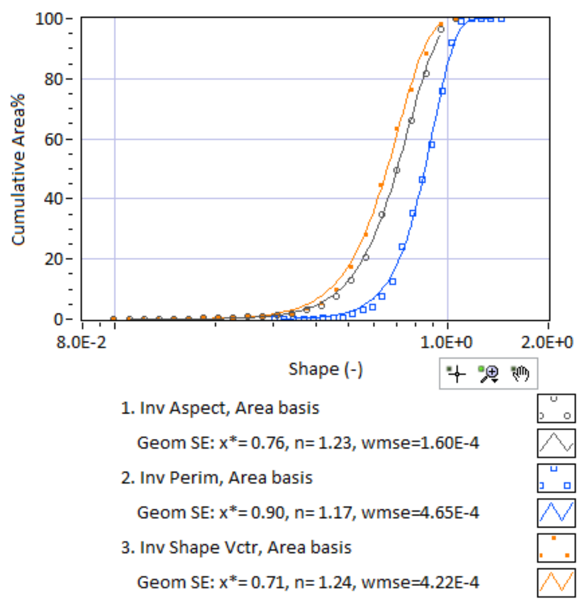

Figure 11. Shape factor analysis suggests that while AR is the stronger component of the overall shape vector distribution, the perimeter ratio (i.e., the irregularity of the perimeter) may be more of a factor with the milk powder compared to the prevous granular samples. Notice the PR distribution has an apparent bias toward low perimeter measurements resulting in higher inverse PR values, some of which are mathmatically illogical (about 15% of the cumulative area distribution, higher on a number distribution).

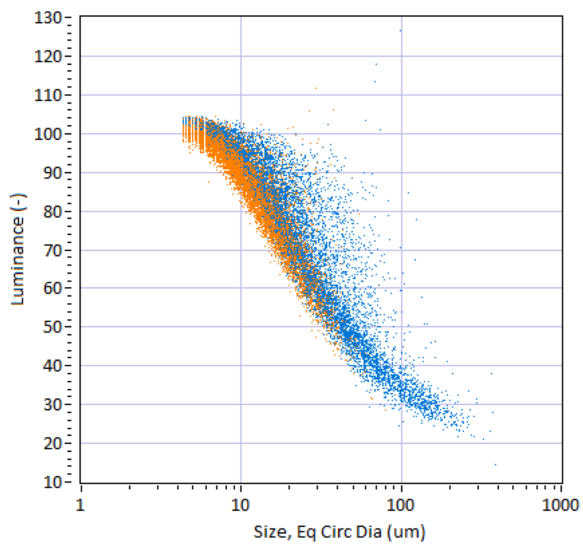

Figure 12. Scatterplot of image data, each point representing one of 20,000 particles. The threshold for image analysis edge detection is at a luminace value of ~104. The blue data have logical perimeter ratios, $PR > 1$; the orange data are mathematically illogical.

Conclusion

Consider Table 1 as an outline for the review, with the content expressed in the above Product/Process Framework, Process Flowsheet, and Sensor Model sections as examples of more detailed content. Please let me know if you have specific feedback or requests.

References

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