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Challenges in the scale-up of particulate processes—an industrial perspective

Timothy A. Bell

DuPont Engineering Research and Technology, Wilmington, DE, 19880-0304, USA

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Abstract

Studies by the Rand Corporation in the 1980s identified substantial differences in the scale-up and start-up performance of plants processing particles versus those processing liquids or gases. These differences were inevitably unfavorable. Particulate process plants take longer to start up and are less likely to achieve desired production rates. Facility operators often underestimate the challenges involved. These problems generally relate to an inadequate understanding of the behavior of particle systems. Many of these behaviors are sensitive to process scale or process history in ways that would not be expected by engineers familiar only with liquid or gas systems. Empiricism must often substitute for first principles. Modeling provides some answers, but often not enough to eliminate the need to operate pilot plants. This paper reviews some of the unit operations involved in particle processing and highlights scale-up issues involved. The use of information from suppliers and other third parties is discussed, as well as scale-up strategies in competitive or regulated industries.

Keywords: Scale up; Start up; Process plant; Merrow; Rand; Pilot plant; Particle processing

1. Introduction

The scale-up of particulate processes has been a challenge since the advent of the industrial age. Processes that were once performed by hand and guided by experience were enlarged by simply increasing the size of the equipment. Problems naturally occurred. However, unlike many similar situations in chemical or mechanical engineering, the problems in particle processing were often *not* resolved through the development of a fundamental understanding of the underlying physical phenomena. Useful equations such as the ideal gas law ($PV=nRT$) in chemistry have few counterparts in particle technology. In many cases, solutions to scale-up problems were developed by trial and error, a practical approach but one that has the unfortunate consequence of dooming each generation to repeat the experiments of its ancestors. While our empirical and computational methods have become more sophisticated, there is still a remarkable lack of “first principle” methods to design particle processes. Merrow [1] aptly describes this as the “theoretical poverty of solids processing”. Progress has been limited by the widespread perception in industry that there are no fundamental principles for some aspects of particle technology, leading them to conclude that research is pointless and trial and error approaches are required [2]. There is a curious resignation to the status quo on the part of many plant operators. The general ignorance of particle technology in industry will cause some to simply hope that problems will go away instead of searching for reasons for unusual phenomena.

Particle technologists often work within their own specialties, such as the unit operations of particle formation, solid/liquid separation, and solids handling. Each may try to optimize his operation. However, a synergistic view of the entire process is required in order to achieve business success. For example, no business enterprise would want a highly efficient crystallization process that generates fine, fragile crystals that cannot be de-watered and will break up into fines in downstream handling. Fig. 1 illustrates the progressive degradation of particles that can occur as they pass through the unit operations of a crystallization plant.

Globalization and stagnation in some industrial sectors has placed mutually exclusive goals upon business management. There is a new emphasis on efficiency, and simulta-

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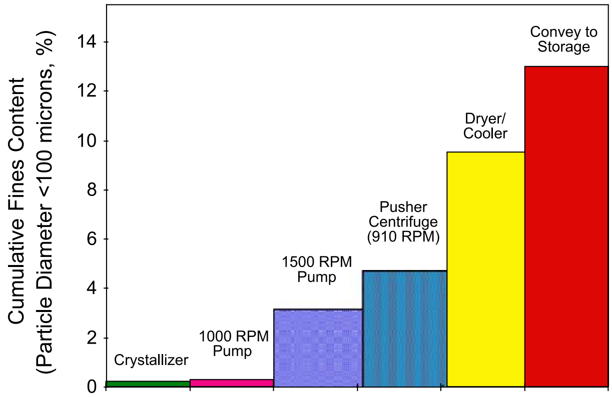

Fig. 1. Progressive attrition of crystals during processing and handling. Data provided by Ross Kendall of DuPont from original documents by E. Kratz and F. Hoyer of Escher Wyss AG, Zurich, Switzerland.

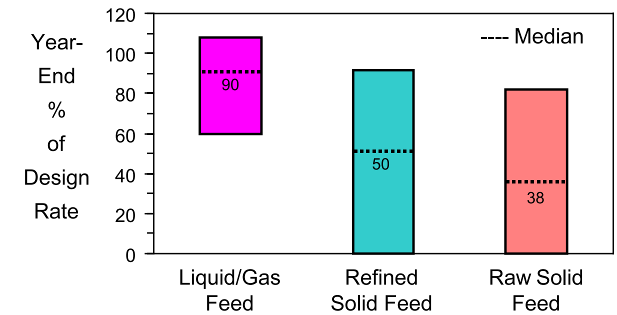

Fig. 2. Influence of feed material on the performance of new plants. Note that some plants produced no product in their first year, and all may have contained some new technology (from Merrow [1]).

neously great pressure to reduce cost by cutting operating staff and plant project teams. These groups are often the repositories of unwritten empirical knowledge about particle processes. Existing plants are often running at full capacity, leaving no time for basic experiments that may provide data for new plants. At the same time, many industries are focussed on the growth that can only come from new products, shifting research resources away from fundamental understanding and optimization of existing processes.

2. The Rand and Merrow reports

In the early 1980's, The Rand Corporation, a private research organization, studied R&D needs in solids processing. The work was sponsored by the United States Department of Energy and a consortium of oil and chemical companies. It followed a period of significant difficulties in the start-up of new synthetic fuels plants. The Rand Corporation reviewed the performance of 37 new plants, using data provided by 25 companies.

One of the Rand study authors, E.W. Merrow, described the work in a landmark 1985 publication [1]. This publication, detailing the poor performance of solids processing plants, is familiar to anyone who either writes or reviews grant applications for academic research in solids processing. The 1985 article and Merrow's subsequent publications [3,4] should be required reading for industrial management and academics involved in particle technology. While Merrow's publication in a popular journal attracted great attention, some of the underlying technical and philosophical issues were concurrently identified in a chemical process scale-up book by Bisio and Kabel [5].

The 1985 Merrow article [1] reported that there was a strong relationship between plant feedstock type and the production rate (as a percentage of design rates) achieved by the end of the first year of operation. Fig. 2 illustrates the data. Liquid/gas feedstock plants performed better than refined solid feedstock plants, which in turn were better than raw solid feedstock plants. The latter plants achieved less than 40% of capacity in their first year. This topic was revisited by Merrow in 2000 [4] using a much larger, more recent database (508 plants, 1996–1998). The trends (Fig. 3) were the same, although performance across various types of plants was generally better, particularly for raw solid feedstock plants, which rose to 77%. However, the more recent plants may have contained less new technology than the pioneering plants in the first study. Merrow reports on the adverse effect that new technology has on plant operability [1,3,4] and demonstrates a strong statistical relationship between the number of process steps with new technology, the starting feedstock type and the startup time and production rates for new plants.

The effect of new process steps on start-up times is frequently underestimated, as shown in Fig. 4 [3]. While plant operators may allow extra time to start up one new process step they tend not to allow multiple units of time for processes with multiple new steps, believing that the start-up issues can be addressed concurrently. This reflects some lack of understanding of the nature of particle processes, in which the performance of each stage of the process is determined by the preceding one. Plant processing steps must be started

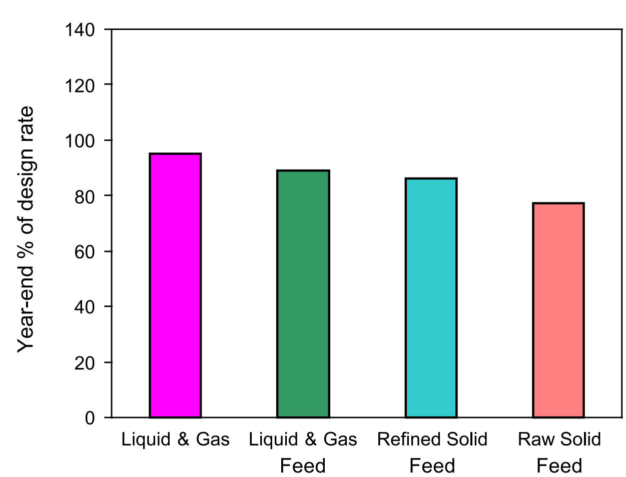

Fig. 3. Performance of new plants as function of feed material. Plot depicts median results (from Merrow [4]). Presence or absence of new technology in the plants is not specified.

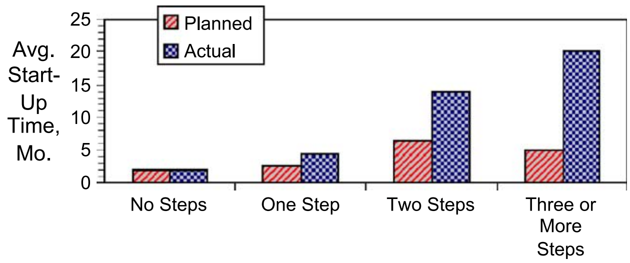

Fig. 4. Start-up time versus new process steps (from Merrow [3]). New process steps are defined as having never before been used commercially.

in sequence, and it is often not possible to fine tune (or even operate) one step until the preceding one is optimized. For example, an out-of-control precipitation process may produce particles that cannot be properly dewatered, generating unmanageable slurry feed to a downstream mechanical conveying system intended to transport solids.

Merrow has also demonstrated a strong relationship between the process designer's knowledge of heat and material (H&M) balances and the start-up performance of a new plant [1,3,4]. He identifies H&M balance data as critical to the sizing of every component in the plant [1]. H&M balance data information can come from theoretical understanding of the fundamental process or actual operating data from an existing operation or pilot plant. Given the lack of theoretical understanding of many particle processes, it is inevitable that there will be a heavy reliance on operating plant data for plants that duplicate existing technology and on pilot plants for first-of-a-kind-process steps.

3. Innovation and pilot plants

Merrow's publications rightly stress the importance of pilot plants, and make a strong case for large scale, fully integrated pilot plants using the *identical* process components as intended for the final plant [4]. Similar advice (although with less emphasis) can be found in Bisio and Kabel [5]. Given the low plant productivity and long start-up times associated with not taking this advice, one has to wonder why anyone would build a new plant without fully piloting the design.

This question is at the center of many capital project decisions and figures prominently in the post-mortem of failed projects. There are three probable reasons for not piloting a process. The first is ignorance of the work of Merrow and others. Those reading this paper belong to the minority of plant operators and designers who follow the literature. Despite the nearly 20 years since Merrow's 1985 publication, many are not familiar with it.

The second cause is pride. Merrow's data represents averages and most engineers consider themselves above average. Many of them believe they are clever enough to beat the odds. In some situations, there is also a career reason and peer pressure to take a chance. A cautious engineer who insists on piloting his step of the process may appear to be non-cooperative or lacking in self-confidence when the rest of the project team is willing to proceed without pilot experience. There is also a human element at play. Most engineers like to improve things. Many are not content with something that is functioning poorly. They would be happy to experiment with an integrated pilot plant. When budget constraints preclude the construction of pilot plants or parallel processing with new technology, the only outlet for the engineer's creativity is the design of the new plant. The situation is aggravated when an idea that should have been evaluated in an experiment becomes a “certainty” during the plant design.

The third reason not to pilot new processes is a legitimate need for innovation and a haste to get new products or processes to the marketplace. The construction, operation, and refinement of a pilot plant will take many months and can cost millions of dollars. Business management may conclude that a business opportunity window will no longer be open if the process development timeline is too long. While Merrow convincingly illustrates the risks, engineering and R&D management is under intense pressure to collapse timelines and show some immediate profits from their work. Unfortunately, collapsing timelines can produce the opposite result of long, very expensive start-ups of full size plants when the same or better overall timing could have been achieved at far lower costs via the use of pilot plants.

The culture and history of a company can influence how it approaches a scale-up situation [6]. When combined with business imperatives that may differ from one company to another, seemingly similar problems may be approached in different ways by different companies in the same industry [5].

For a business to make an informed decision on the operability risks of a new plant, it must be able to list the adverse events that may occur, assess the likelihood that they will occur, and estimate the severity of each event. This technique, known as Failure Mode and Effect Analysis (FMEA) is widely used in safety and quality studies [7], but is more broadly applicable. For example, a new dry grinding and classifying plant may suffer plugging problems, unacceptable wear life of components, and failure to produce the required particle size distribution. Each of these events has some likelihood of occurrence, and the consequences or magnitude of the occurrence could be estimated. A competent project team can probably list most (but perhaps not all) of the possible events. Checklists can be useful in such an exercise. Estimating the probability of occurrence is much more difficult. In addition to the technical unknowns, there can be a tendency of a project team to understate the likelihood of occurrence, since to admit otherwise may be an acknowledgment of some inadequacy of their work or its premises. Finally, participants in such a discussion may be too optimistic about the potential severity of such problems. In any such discussions, it is prudent to plan a response to the adverse outcomes. Anticipated serious problems for which there is no viable "fix" are a serious warning sign for the project team and an indication that piloting of the process is required.

The only way to avoid the use of pilot plants is build every plant as a clone of its predecessor. Indeed this is often done in the pharmaceutical industry where time-to-the-market and regulatory constraints typically outweigh plant productivity concerns. Similarly, Intel, the giant integrated circuit manufacturer, clones its US$2 billion plants all the way down to the selection of building paint colors [8]. The strategy improves product quality and productivity, but may stifle innovation at the plant level. In this case, innovation must come from the next generation of chip manufacturing technology with development centrally managed.

Duplicating existing particle processing plants provides opportunities for the development of functional excellence across an enterprise. Lessons learned at one plant can be immediately applied to the others. In ideal cases, this could lead to a company becoming the low cost producer in an industry. This presumes, however that the feed materials to the process are consistent, knowledge is shared freely amongst plants and that the basic equipment in the process train was optimally chosen initially. It also presumes that the products to be produced are consistent in quality and chemical nature from year to year. While these criteria may be satisfied in some raw mineral and basic polymer processing plants, they rarely occur in higher value chemical and polymer operations. There are too many changes likely in the supplier and customer marketplace, and it is probable that the design of the first plant was not optimal to start with.

If the first plant using a new technology does not run well, it would seem obvious to the owners that cloning it would not be a good idea. Indeed, Merrow states "It is extremely imprudent to begin commercial design of a second version before the pioneering design has been proven" [4]. However, plant operators may delude themselves into thinking that after some months (or years) of operating a poorly performing plant that they know precisely what it would take to fix the next one. Hence, they feel quite capable of skipping the pilot plant verification of the design for a new plant. However, in actuality, they may not know how the proposed change would work by itself, or what effect it would have on the downstream process.

There is no real answer to this problem. Merrow and the bitter practical experience of industry demonstrate that the failure to build and operate integrated pilot plants will cost time and money. However, in all but the most basic industries, the pace of the marketplace and the impatience of investors will encourage the taking of risks. The only way for technologists to manage these pressures is to continuously gather data from existing processes, and to have the courage to admit (or even argue) that they do not have sufficient information to proceed with a new design.

4. Why particle processes are difficult

Merrow's data makes it clear that it is more difficult to start-up or scale-up a particle process plant than it is for plants handling liquids and solids. He addresses some of the issues in his first publication [1] in which it is noted that R&D expenditures have historically been misdirected—too much of the available money has gone to technical areas that are not the real problems in practical industry. Furthermore, he finds that some of the management involved in R&D planning believe that plant operability issues are a low priority that can be addressed by technicians after start-up. Often the technical interest in a new process is the product itself or a novel new process step, and all other process considerations are secondary.

This neglect of process technology has consequences that are more painful in particle operations than in others because of inherent differences between particles, liquids, and gases. Particles can almost be described as a fourth state of matter (after solids, liquids, and gases) since they can take on the behaviors of one or more of the other states. For example, dry particles in a certain size range in a silo can develop cohesive strength and transfer shear stresses like a true solid. Most particle assemblages demonstrate significant compressibility and some are also elastic, like a gas. While discharging from the silo, they can retain air and take on liquid-like properties to the extent that handling equipment cannot regulate the flow. The behavior of a particle system is more likely to be inconsistent than consistent. It may depend on every event that ever happened to the particle from its creation to its ultimate consumption. Unlike gases or liquids, particles often remember where they have been and never forget.

Continuum theories simplify the life of engineers dealing with liquids and gases. Fundamental laws can be applied equally to volumes ranging from tiny to huge. Time can be a factor in reaction rates, but many of these rates can be measured on the bench scale and scaled up as needed. Chemistry that works on a small scale will work on a large scale, and unlike particles, liquids and gases do not have cores and shells that may respond to a process at different rates and require different analytical techniques. Gases and liquids do not grow, agglomerate, aggregate or suffer attrition, but particles do.

This sensitivity to history and scale makes it hard to quantify particle systems. A characterization technique that is applicable to one stage of a process may not be accurate or relevant in the next stage. Understanding a particle process may require exhaustive characterization efforts. Particle formation techniques such as crystallization, precipitation or aerosol formation frequently are very sensitive to process scale (i.e. size), so information gathered at one scale might not reliably predict behavior at another. Processing unit operations may also have a significant time elements associated with them. These time and length scales, when considered together, present a great challenge to understanding, modeling, and control. Fig. 5 illustrates the situation for a full-scale crystallization process. While process designers will attempt to duplicate pilot process conditions on scale-up, they may be unaware of which conditions are critical to reproducible behavior. They will frequently try to push the edges of the design to increase production rates or reduce costs. Bench scale experiments often use equipment such as filter paper for dewatering, vacuum tray dryers, and manual materials handling that generate unrealistically good results and defer discovery of operability problems.

The scale-up of processes using the principles of similarity quickly encounters practical and theoretical conflicts. Simultaneously maintaining similarities in equipment shape, velocities, power inputs, etc., can be impossible, forcing the designer to choose which factors are more important based on experience with the process [9]. For a new process, this experience may be unavailable. Even when there is such experience, it is not possible to include particle size as a factor in similarity calculations because it does not change with process scale.

An example of this problem is the scale-up of stirred vessels containing particles in a liquid. Such vessels are widely used for crystallization, precipitation, fermentation, and other operations. A comprehensive literature study by Geisler et al. found a broad range of correlations intended to prescribe the required stirrer power input for a full size vessel based on data obtained from laboratory scale units [10]. Geisler found that most of the empirical literature was developed using too narrow a range of operating parameters, in vessels that were too small. This led to correlations that proved to be specific to a narrow range of vessel diameters and particle sizes rather than broadly applicable. Geisler ultimately concluded that there are no simple and constant scale-up rules for stirred vessels, since the actual power required will depend on many factors, including the particle and fluid properties as well as the vessel diameters. Similarly, Wypych reviewed existing correlations and found wildly varying minimum conveying velocities for pneumatic transport as a function of particle size and pipeline diameter [11].

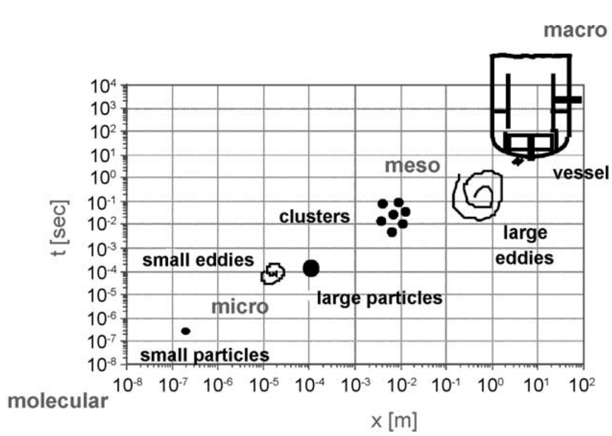

Fig. 5. Range of time and size scales encountered in crystallization processes. Graphic provided by Daniel Green of DuPont.

Geisler [10] also reported on hysteresis in stirred tanks, in which the conditions necessary to maintain a suspension were not the same as those needed to create it. A similar situation occurs in pneumatic transport, where the saltation velocity (the gas velocity below which a particle will drop out of a horizontal system) is lower than the velocity necessary to pick up a particle off the floor of the pipeline [12]. Hysteresis is also known to occur in the distribution of stresses in a silo depending on whether the silo is being filled or emptied [13]. Recent research [14] further complicates the problem by showing that the ratio of horizontal to vertical stresses in a silo is not a constant across the silo cross section and can change markedly as a result of repetitive filling and emptying cycles. These examples of hysteresis are typical of particle technology, and make it difficult to ever be sure that a particular phenomena is fully understood and that the results from the literature include all relevant factors.

While existing processes would seem to be an unbeatable source of process information, extraction of such data from the process can be extraordinarily difficult. Accurate measurement of in-process parameters such as temperature and chemical composition can be problematic in processes where fouling and erosion confuse and destroy instruments. Some operators give up on maintaining instruments and do without. Very few processes are properly designed for the acquisition of valid in-process data or samples, leading to suspicion of segregation or process lag time in whatever data is acquired, especially for dry particles. More often, a decision is made that a particular step cannot be monitored, and guesswork is substituted for data. Even when such data is available, careful stewardship of the resulting information is rare, particularly in operating cultures where the process is considered to be black box with a mind of its own. This situation is a particular tragedy, since the widespread availability of data logging and statistical software makes it possible to mine plant data for previously unobserved trends. It is surprising (and sometimes embarrassing to specialists) how often a process insight is developed as a result of trying to explain a statistical conclusion.

Process equipment in particle plants is much more likely to encounter severe and unexpected stresses than its counterparts in gas/liquid tanks. For example, a tank agitator may be designed for a particular slurry viscosity, only to find that process upsets generate a slurry of much higher viscosity or one that will not stay in suspension, causing high start-up loads. Some processes will combine corrosive slurries or vapors with the transport of abrasive solids, leading to extremely short equipment life until appropriate materials of construction are located or the process conditions are modified [4].

5. Equipment vendors

If theoretical knowledge is unavailable, and a firm is unable to pilot a new plant, they may attempt to test as many of the unit operations as possible in an equipment vendor shop. In fact, even if the firm was reasonably certain of the required equipment specification, they are still likely to run tests in vendor shops to ensure that the vendor will take responsibility for the performance of their equipment.

Tests in vendors shops are better than no tests at all, but sometimes only slightly so. The vendor may be unable to duplicate many aspects of the actual process. As the number of complications (temperature, pressure, toxicity, corrosion, gas flow rates, etc.) associated with a test grow the likelihood of a vendor being willing or able to conduct a realistic test declines quickly. Tests with surrogate materials, or with actual process material under non-standard conditions are of questionable usefulness. The selection of surrogate materials is both a skill and an art, and requires comprehensive characterization data on the materials involved and sufficient judgment to determine which characteristics are critical to a test. Even if a surrogate is found that mimics features such as particle size distribution, it is nearly impossible to duplicate behaviors during mechanical processing such as shear thinning or thickening, wall scale formation or heat transfer rates. Even with a valid test, scale-up of the vendor's laboratory data is often required, since the test equipment may be significantly smaller than that required for a full size process.

These limitations are illustrated by a case history. Dense phase pneumatic conveying was being considered as an alternative to mechanical conveyors to handle a hot, abrasive material at high transfer rates. Tests at a pneumatic conveying vendor shop were conducted at ambient temperature, and the pipe cross-sectional area was about 1/3 that required for commercial production rates. Stainless steel piping with welded and bolted flanges was used in the laboratory, but due to abrasion the full-scale installation would require hardened cast iron pipe with special clamp-type connectors. After the successful vendor test, prudent plans were made to install a dense phase system in parallel with existing mechanical conveyors for extended plant trials. The parallel operation was never conducted, however, due to cost and other priorities at the plant. The dense phase system was instead installed in a critical process train for a new plant. The start-up was a disaster. Massive failures occurred with virtually every aspect of the system that was not (or could not be) tested in the vendor laboratory. For example, line hammer pulsations in the full-size pipe were sufficient to pry apart the clamp-type connectors, permitting abrasive material to leak. The leaks rapidly eroded the flange faces, necessitating replacement. After months of production losses, the system was scrapped and replaced with mechanical conveyors.

For many types of equipment, a vendor will guarantee performance presuming that the material he tested in his laboratory is identical to that in the plant. This creates a false sense of security for project engineers. For new processes, such a guarantee can be an automatic “escape clause” for the vendor if he chooses to use it. It is extremely unlikely that the actual production material will match the test material, which may have come from a pilot plant or a presumed surrogate source. If the equipment fails to perform, the vendor's liability is generally limited to the cost of the equipment. This can be a tiny part of the cost of a delayed plant start-up or poor quality product. On the other hand, it is not reasonable to expect vendors to accept liabilities that could be 10 or 100 times the values of the equipment involved.

Vendor tests could be more useful if both the customer and the vendor were willing to work harder to create a meaningful test. For small pieces of equipment (such as feeders), a vendor may plan to conduct a test of few hours duration during which the best possible performance is often obtained. Such tests may be too short to assess problems such as build-up or abrasive wear, and are usually limited to one or two different samples of material. A process that uses the same feeder to dose several different ingredients may not be properly validated unless every candidate feed material is tested. Vendors often want to minimize the duration of tests in order to limit costs and to free up the test laboratory for other customers. Customers aggravate the situation by being reluctant to send sufficient volumes and types of materials to test. Such material is usually scrapped and is often a disposal problem. Recycling of material through the test equipment will frequently cause changes to properties due to attrition, moisture gain or loss, or mechanical processing. While recycling may be necessary, the final test should always be conducted with fresh material.

The customer may be unwilling (or unable) to provide sufficient details to allow the vendor to design his equipment. In one case, a vendor of hammer mills was asked to provide a mill that could grind lead pellets at a rate of 100 kg/min. The vendor presumed that the feed to the mill would be continuous, and the customer did not inform him otherwise since he felt it would disclose too much about his process. The actual installation delivered chunks of lead weighing 30 kg to the mill every 20 seconds. Equipment failure occurred instantaneously at start-up. While this particular incident occurred nearly 50 years ago, similar problems occur today.

Few equipment vendors can provide fully integrated processes in their test laboratory. The vendor may provide a piece of equipment intended to be placed in the middle of a process, but not have any responsibility (or knowledge) of the upstream and downstream processes. Connections to upstream and downstream processes must be simulated in some way in a vendor test, and since these arrangements are usually unique and temporary, they tend to be crude and may not represent reality.

As many large chemical companies have “downsized”, they have reduced their in-house capability to select and specify process equipment. There is often an implicit or explicit assumption that this role will now be assumed by equipment vendors. One would then expect that the vendors would increase their technical staff in order to supply this service, but this is rarely the case. Purchasers are no more willing to pay extra for technical support than they were prior to the downsizing, so many vendors cannot afford to enlarge their staff.

Industries considering a new process or a process scale-up will often look to equipment vendors for advice. This can be a mistake for the early stages of first-of-a-kind processes when a broad view is required. Vendors will always know their own equipment on an empirical basis. They may know the fundamental theory behind their equipment, although this is not always the case. A vendor is likely to try to scale a new piece of equipment for a new process according to some prior experience with a different product. While this may be necessary, it adds significant risk, particularly if the vendor lacks the characterization facilities and technical skills to determine how the old and new materials relate to each other.

Vendors are not likely to be familiar with completely different technologies for accomplishing the same purpose (such as filter presses versus centrifuges) unless they sell both. The pressure to make a sale forces them to be optimistic about the capabilities of their own engineering and equipment, and few are likely to recommend a competitor for a particular project. More subtly, they may recommend their type of equipment for a project where it is an adequate, but not optimal choice. On the other hand, a competent vendor might decline to bid on a project that he thinks is unworkable. This is always a warning sign to project engineers, and before looking for an alternate vendor, it would be prudent to understand the first vendor’s reservations. Vendors with prior experience in a particular industry will have valuable experience that may be reflected in the design features (and price) of their equipment.

Customers can undermine the potential contribution from equipment vendors by placing projects out for bid (tender) to vendors from other industries whose equipment may not have the design features proven necessary for a particular duty. Savings in the original capital cost must be weighed against future downtime and maintenance costs. For example, the cost of a stainless steel rotary valve can double depending on quality and design details. The cheaper valve may be adequate for many purposes, but the person selecting it must make certain it is suitable for a particular project. This is becoming a larger problem as businesses rely more on outside engineering firms and alternative vendors from less developed countries. Even when the person specifying the new equipment is familiar with an existing equipment installation, he may not know which of the equipment features are critical to the process.

6. Suppliers and consultants

The supplier of an ingredient for a process may be able to provide design recommendations based on experience with previous customers. This advice can be quite valuable, although its availability will vary by supplier and industry. A supplier will also have its own in-house experience producing such a material. However, the production process for a material may bear little resemblance to the process in which it is consumed, so such experience may not be of value. Many suppliers are surprised at how difficult their product is to process after it has been aged or subjected to repetitive handling.

Private consultants or university professors can be an excellent source of technical guidance for specific unit operations within a particle process. Unlike equipment suppliers, a consultant can be expected to be familiar with all the technologies that are relevant to a unit operation and can be used to guide technology selections. They are often restricted in their knowledge of upstream or downstream process steps due to their own technical limitations or their client’s reluctance to disclose the process details. Consultants with expertise in certain industries (rather than unit operations) may be able to serve as a sounding board for an integrated design proposal but may not know alternative technologies, especially for totally new processes.

Probably the greatest limitation of consultants is industry’s unwillingness to use them appropriately. Too often, the consultant is not brought in until the plant is built and the troubles have started. This may be too late for anything other than stopgap measures. While some processes can be corrected after they are constructed, far more particle-based processes cannot readily be fixed if the wrong equipment is in place. Merrow [4] has quantified the beneficial effects of bringing in specialists early but does not dwell on why they are often brought in late. As with pilot plant decisions, the reasons are typically economic or political. An engineering design firm or plant technical staff that prides itself on its competency may be reluctant to admit that they do not have the resources to design each step of the process. Allocating some of the work to consultants may deprive a design firm of needed revenue. Consultants may contribute to a perceived-value problem by being unwilling to take a definitive or unpopular position for fear of losing the client.

7. Challenges within unit operations

It is not possible to discuss every unit operation in particle processing in a brief article like this. However, a few observations from several common operations (crystallization, centrifugation, grinding in media mills and silo design) can illustrate the difficulties in industrial scale-up.

7.1. Crystallization

The production of crystalline particles via wet crystallization is a mainstay chemical process. Crystallization processes are notoriously difficult to scale up, leading to long start-up periods or unmet particle size, purity, or production rate requirements. The performance of a crystallizer entails the interaction of many factors such as solubility and supersaturation level, nucleation and growth rates, particle size distribution, impurities, solids loading and settling rates [9]. Most of these factors interact with each other in a non-linear way, leading to a design problem of remarkable complexity. Complete data on these phenomena is rarely available. Furthermore, these factors all interact on the micro scale around each particle [15] such that in any vessel without perfect mixing, the processes may be occurring at different rates or in different ways at various locations. Since no full-size vessel is likely to have perfect mixing, true optimization of the entire process may be impossible.

One of the reasons crystallizers are difficult to scale up is because they are difficult to scale *down* accurately to a pilot scale, particularly for continuous operations. As crystallizers become smaller, temperature control becomes problematic, and key factors such as feed injection point(s), residence time and the frequency of passage through high shear environments (such as agitators) become grossly different from full scale [15]. Common full-scale problems such as encrustation of heat transfer surfaces do not occur, or occur at different rates in different places. It may take more time than expected to reach steady-state operation, and if oscillations of particle size distribution occur, the unwary experimenter may be misled. Finally, it may not be possible to precisely duplicate the impurities that can be expected in recycling liquor in the full-scale plant. Such impurities may markedly change the crystallization behavior and the resulting crystal purity [9].

7.2. Dewatering in centrifuges

Centrifuges are commonly used for the separation of particles from liquids. While one thinks of a centrifuge as a single piece of process equipment, a centrifuge will actually perform cake-forming, washing, and dewatering operations. These processes may occur sequentially in the case of batch centrifuge operation or concurrently in a continuous centrifuge. Dewatering behavior is affected both by equilibrium parameters (how strongly the liquid is attached to particles) and by kinetic considerations (how fast liquid can be moved through the particles). The combination of these factors determines the efficiency of the centrifuge and its throughput. However, the dewatering behavior is also influenced by the nature of the filter cake that was formed prior to dewatering and the uniformity of particle size throughout the cake. These parameters can combine such that the throughput rate may have to be reduced by a factor of 100 in order to achieve a small, but important reduction of residual moisture. Some filter cakes may be compressible (reducing their permeability), or crack so that washing operations are largely ineffective [16,17].

Keller [16] lists 6 basic factors (such as particle size and liquid viscosity) affecting centrifuge selection and notes that many such factors can have values over a range of several orders of magnitude. This has led to the development of a plethora of design types of centrifuges (14 in total per Keller) and many variations within each design, such as 15 types of decanter centrifuges. Fig. 6 illustrates some of the choices available and presents the confusing multitude of choices for

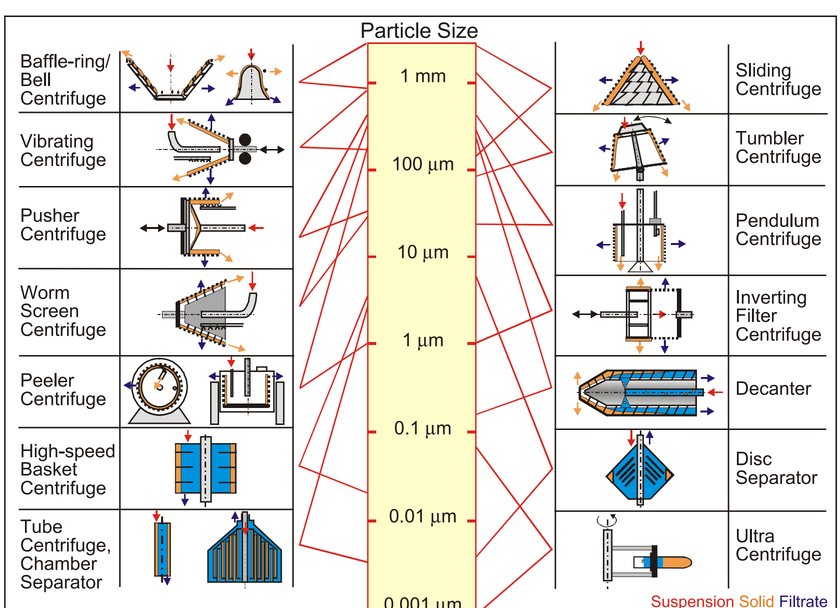

Fig. 6. Centrifuge selection alternatives and possible correlation to particle size. Graphic provided by Karsten Keller of DuPont.

dewatering particles of a specified size. Simple tables or decision trees are insufficient to either select a piece of equipment or predict its performance because the properties of the solid/liquid system vary throughout the centrifugation process and are difficult to characterize. No vendor carries more than a few equipment types, and few can deal with hazardous or age-sensitive liquids in their test laboratory. Decision-making based on an end-use performance attribute (such as residual moisture content or throughput rate) does not provide any insight as to optimization possibilities within the machine. Extraction of insitu performance parameters requires expensive and exotic equipment such as a laboratory centrifuge fitted with telemetry sensors [17]. Such equipment is rarely available in industry.

7.3. Grinding in media mills

Wet media mills are a popular tool for fine grinding and dispersion of particles in the size range from nanometers to a few micrometers. As with any grinding task, the system designer will want to know the smallest particle size distribution that can be obtained, and the amount of energy required to produce the required particle size distribution. This data is a material property that cannot be obtained by study of the mill itself. Careful studies by Schwedes and his coworkers (including Kwade [18]) have led to a good understanding of media mills. Kwade identifies two essential factors (the number of stress events and the stress intensity) that govern the grinding process. Some particles fracture due to many small impacts, while others require a single force of large magnitude. He reports that the occurrence rate of such factors in the mill is a statistical distribution based on mill geometry and residence time distribution. For a given mill, grinding media and slurry viscosity it is possible to develop a useful curve of particle size as a function of grinding time (i.e., the number of stress events). From this, the performance of that particular mill can be predicted for that particle system. The specific energy (KW/ton) required to produce a median size can be determined.

Problems arise when a milling process is to be scaled up. The specific energy determined on the laboratory scale reflects the product of the number of stress events and the stress intensity as well as mechanical inefficiencies within the mill. Use of a larger mill to deliver more energy (and hence higher throughput) does not ensure that the proportions between stress events and stress intensity stays the same as it was in the laboratory mill. Depending on the particle system involved, the grinding performance might improve or deteriorate. Surprisingly, it is not possible to directly predict the energy that will be drawn by a mill. Installation of a larger motor does not ensure that the mill will deliver more energy. As media mills get larger, the power density (kW/liter of mill volume) will go down, sometimes by a significant amount. This can lead to a gross underestimation of the number or size of mills required, even if the total required energy input is known.

7.4. Silo design

The design of silo hoppers and outlets to prevent arching is well established [19]. However, for silos storing cohesive materials in which the hopper design and material flow properties do not permit a mass flow discharge pattern, the silo operator must contend with the possibility of ratholing. (A rathole is a type of self-supporting flow channel through stagnant material, leading to incomplete emptying of the silo). Ratholing problems can be overcome by the installation of flow promotion devices or by enlarging the silo outlet to a point where the rathole is no longer stable, known as the critical rathole diameter. While Jenike [19] provides two formulas for the calculation of the critical rathole diameter, they will rarely agree and can be very conservative depending on the silo size and other parameters [20]. Conservative estimation of the rathole diameter results in excessive cost for discharging devices and nonsense results such as stipulating that the silo outlet must have a larger diameter than the silo itself. Unlike most of the issues discussed in this paper, this particular scale-up problem may be solved in the future by mathematical modeling (independent of a specific material) and one can hope that improved equations can be developed. The challenge in this case is the shortage of researchers. The number of world-class investigators in silo flow issues is declining, as senior faculty members retire and new academics are drawn to fields that are more glamorous.

Flow promotion devices such as vibrators, air cannons, or air injection systems are routinely used in cases where the material being stored has poor flow properties. Even when the silo designer is aware of the flow problems, he may need to use a flow promotion device to cope with an undersized silo outlet dictated by downstream equipment. There is virtually no established theory that would lead to the effective equipment selection or sizing of flow promoters [20]. While on the pilot scale a process operator can apply extraordinary manual flow promotion methods, such methods become ineffective on large structures. Flow promotion equipment vendors will generally take a “try it and see” approach, and provide empirically based sizing criteria that are based on subjective judgements of the underlying flowability problems. When compared to the cost of major process equipment, flow promotion devices are cheap, but their operating costs and consequences such as noise, metal fatigue, and compressed air usage can be significant. If a flow promotion technique is completely ineffective at start-up, the process is likely to come to a stop until the problem is fixed.

8. Modeling

The largest amount of academic enthusiasm and probably the greatest actual progress in recent years has been in the field of numerical modeling of particulate processes. Many

PhD studies now have a modeling component even if the principle work is experimental. Some in industry may wonder if traditional studies have been sacrificed in order to make room in the PhD course for modeling, but the net effect has certainly been positive. There are many industrial scale-up situations in which modeling is the only way to develop an understanding of a complex phenomenon or one in which physical measurements are difficult or impossible.

Three modeling techniques, Discrete Element (DE), computational fluid dynamics (CFD), and Population Balance are commonly applied to particle processes. Finite Element (FE) methods are selectively applied. Each technique has its specific applications, and each requires assumptions or data that can be difficult to obtain. Models with 20 variables are common and in extreme cases, up to 200 variables may be identified [21]. The quality of the assumptions or data is in many cases the principal limitation of the model, and diligent modelers are generally anxious to obtain data that can be used to validate their work. Unfortunately, complete validation is rarely achieved, since in most cases the modeling is used to study a problem for which physical measurements are difficult or impossible. Of the three modeling techniques, CFD has by far the greatest use in industry, perhaps because the same concepts have been used for gas/liquids flow for years and commercial software is readily available and widely supported. Commercial software is also starting to become available for DE modeling, as well as integrated DE and CFD models [22].

The accuracy of a model is in part determined by how far the modeler had to extend beyond what is known from measurements via physics. FE models (used for stresses and displacements in packed beds) can be reasonably accurate predictors of stresses if the bulk properties used as constitutive parameters are measured. DE models when used for the same problem require the mechanical properties of individual particles, which can be impossible to obtain. These properties are then extended via physics into theories of bulk behavior. Even when the particle properties are specified, various DE research groups utilizing different techniques may calculate stresses that differ significantly from each other [23]. The finite element techniques, on the other hand, struggle with boundary conditions where the bed is no longer packed. Traditional DE modeling is limited in its consideration of the shape and properties of particles. It is difficult to explicitly consider the effects of fluid flows. Conversely, CFD generally neglects interactions between particles, a particular problem in dense flows.

These restraints illustrate the scope limitations of traditional models. Most modeling concepts will focus on a particular length scale or time scale, and rely on other information (or other models) to provide the tie-in to smaller or larger scales. This is particularly important in particle processes for which the past, present, and future of the particles are completely intertwined and interdependent. Fig. 7 illustrates a grinding circuit, perhaps one using a fluid energy mill. DE modeling may be used to study the collisions of individual particles within the mill, while CFD visualizes the air currents sweeping the particles through the mill. Population balances may be used to examine the particle size distributions and throughput, particularly in a grinding circuit where a recycle stream is present. Finding the appropriate ways to link these techniques is a topic of rapidly increasing interest [24]. These linkages are vital to establishing a

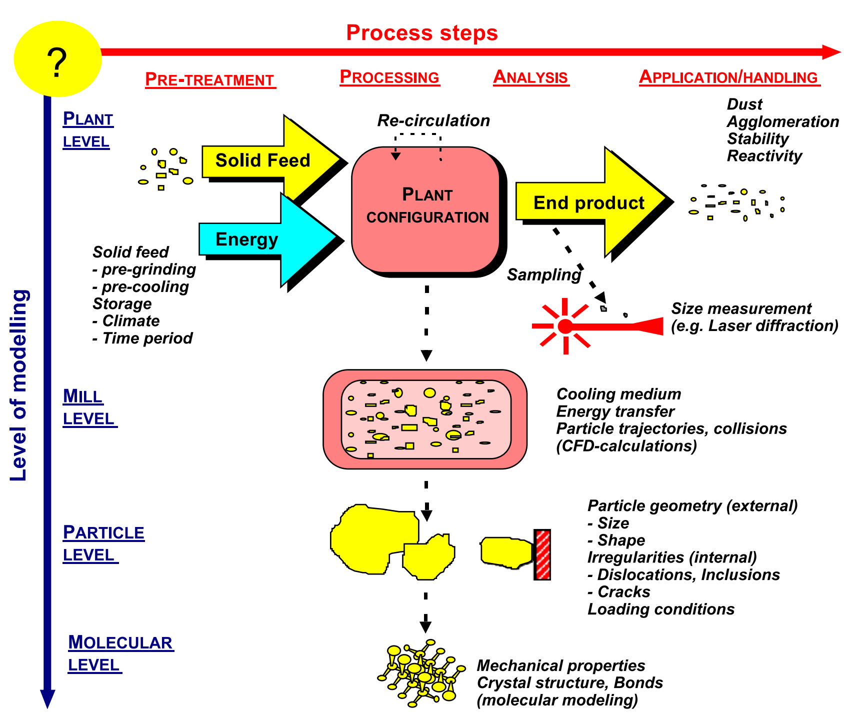

Fig. 7. Application of modeling to a grinding circuit. Graphic provided by Erik Gommeren of DuPont.

useful tool for understanding complete processes, since a single-scale model of only one part of the process will leave questions unanswered or overlook important input or output interactions.

In order to manage computational requirements, all modelers will apply simplifying assumptions to the physical system. Selecting which assumptions to use requires both extensive experience and good practical judgement, skills that may not be found in every person working in this relatively new field. Errors in such assumptions may not be obvious. Mathematical techniques to reduce computation time may require further judgements, such as a decision as to whether it is sufficient to know the number of particles and their powder surface area but not the actual particle size distribution [21].

From an industrial perspective, modeling has three major limitations. First is the time required to construct and run a model. While computation speeds are increasing rapidly, much of the increased capacity is used to run models that are more sophisticated or multiple cases. Quality may be improving more quickly than delivery time. A second problem is the scarcity of skilled practitioners. True expertise in the techniques resides in specialists rather than process designers. This limits the application of the tools. The third limitation is the number of seemingly impractical assumptions entailed in many of the models, which sometimes causes experienced engineers to be skeptical of their value. These individuals may become converts after seeing the first good application of the tools, particularly if the modeler has taken pains to collect their practical input. In general, however, the use of modeling is still limited in process scale-up. This may be because the preceding obstacles are severe enough that only the most critical problems are judged important enough to approach via modeling. It is not yet possible to design a process from modeling alone.

9. Conclusions

The work of Merrow has illustrated the challenges involved in the scale-up of particulate processes. The use of pilot plants, while eminently logical, increases perceived cost and seemingly slows progress. Their value is often not perceived until a failure has occurred. There is ample evidence that particle processes are more difficult and good reasons as to why. Particle systems are hard to characterize or to fully understand, and their interactions with process equipment can be unpredictable. Too much reliance has been placed on equipment suppliers or third party design firms without ensuring that they have the resources to perform the task. Mathematical modeling has high potential, but is still too abstract and difficult to apply for many problems.

The path forward is not altogether clear. While incremental progress is being made on many fronts, progress is generally slow except in the field of modeling, which arguably has much farther to come before it becomes useful. The generally slow progress is understandable in light of the complexity of particle systems, which greatly complicates the development of broad theories. One of the few bright spots is the explosion of data collection and sharing techniques within an experiment, plant, or academic community. Broader dissemination of available data, as well as statistical techniques to process data, will ensure that fewer experiments have to be repeated, and more value is obtained from each one.

Individual engineers working in industry now have access to more data and research than they can use. Study of the existing literature will make them better observers of their processes, and identify areas for research. It may also identify opportunities for industrial-academic collaboration, which does not happen often enough in full-scale facilities.

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