## Think process before scaling up

Tom Mroz, Director of Technology at thermal processing company Harper International, explains the importance of considering scale-up requirements for thermal processing early for successful commercialisation of new materials.

o one can deny that materials developers ultimately have their eye on commercial uses. However, the scale-up process is sometimes ignored in their vision for future applications. So, when is the right time during materials development projects to consider commercial scale issues?

- As early as initial conceptual experiments?
- During initial scale-up of promising results?
- During process refinement and capability determination efforts?
- When market potential confirms the need to begin continuous manufacturing?

As the market for new-materials development expands and time-to-market can make the difference between success and failure, the answer is all of the above.

Certainly, the concept of planning for success is not new – plenty of self-help gurus have made their livelihoods teaching people to do just that. But often in material process development projects, the application of the lesson is delayed or forgotten altogether. To neglect this can lead to delays in project completion, excess costs associated with painful late process changes, or even elimination of a candidate process after significant investment. Even if a project successfully launches, late adoption of scaling considerations can require re-engineering of the process, or missed opportunities for product quality, cost or throughput.

Consider the case of early conceptual experiments. One might start down the path of curiosity, seeking to determine if a particular set of raw materials, process conditions or energy inputs can lead to a particular result. Assuming the experiment is not theoretical in nature but rather directed towards an eventual product offering, care must be taken even at these early stages to consider variables that may be affected by scaling. If commercialisation is the end goal, even the most initial experiments can be screened based on their potential for scalability. For

Above: Testing and sampling of materials is critical, especially to those that require high levels of uniformity and purity.

instance, considerations regarding raw material cost, quality, physical characteristics and other variables that are often neglected at this stage can play an important part in project success in later stages.

Involvement with key suppliers at an early stage can be a critical factor. If involvement with the customer occurs well into their development process, where many variables have already been established, opportunities for optimising cost savings, product quality or throughput may be missed. In the most extreme cases, it can make the difference between success and failure for the project.

## **Getting warm**

Scaling of thermal processes is rarely a simple matter of linear extrapolation. At experimental levels, the conversion rate of many solid-solid and solid-gas reactions is primarily a function of the size of the reactants and the quality of their mixedness. As the reaction is scaled up, the ability to heat or cool the mass of material and the ability to introduce or remove gasses to or from the solids, plays an increasingly important role in reaction efficiency, often to the point of controlling the conversion rate. Careful consideration of raw material forms and process steps can have a big impact on the range of processing options available for later thermal processing methods.

The most efficient thermal processes are engineered to apply heating and cooling to minimal loads. In a perfect situation, this means processing

the reactants in a continuous manner without need for material containment. In a nearly perfect example of such a process, carbon fibre is produced by drawing the material continuously and directly through the various thermal process steps. Energy inputs are required only to heat the fibre and the process cover gas as it enters each thermal step. No secondary containers are required – the fibre supports itself throughout the process.

For bulk particulate materials, other means of manipulating the material in the thermal steps are required and options such as rotary furnaces, fluidised beds and vertical furnaces provide efficient processing options. In each case, consideration needs to be made to the form of the reactants so that they provide the reaction kinetics, as well as having a complete physical form compatible with the process method.

Often, if consideration for continuous processing is ignored until the basic process and materials are fully established, the conversion of the process to suit the desired equipment solution can lead to a long and expensive development project. Earlier consideration and testing of commercially-relevant process methods can minimise these scale-up costs. The result is typically better control over the project schedule, a better and earlier understanding of the costs and facility requirements for the commercial process, and even quicker path to producing process-relevant samples for internal assessment and customer sampling.

For the purpose of inserting production-scale considerations in process testing, Harper International operates a Technology Centre where clients can work with specialists using commercially relevant processing tools. The interaction may involve initial feasibility testing of a concept, scaling of a concept to a continuous operation, or engineering studies to determine the most suitable manner to efficiently conduct a specific thermal process. Later in the development process, it could evaluate pilot scale operations, preparation of test materials for sampling, or construction of pilot equipment for customer use on their own production floor.

When using commercially relevant processing tools, the benefits are not only in addressing the immediate experimental goals, but also in the opportunity to access the equipment design and scaling expertise of your partner's technical staff. The availability of experimental data from laboratory testing provides a strong basis for our design of full-scale equipment. Because of this, the customer can expect the design to closely match his requirements – risks associated with under-engineered solutions or excessive expense are minimised by the ability to use actual process data to inform decisions on scaled designs.



Above: Designing the most effective thermal processing solution for the application must include early consideration for energy efficiency, product quality and throughput.

Below: Continuous carbon-fibre processing in a series of thermal steps. This well-refined process is engineered to apply heating and cooling to the most minimal load possible and without need for material containment.



## Making it work

An example of process overview to aid scale-up is the reduction of molybdenum from the oxide to metal which occurs in several steps, where the initial reduction from trioxide to dioxide is exothermic. The ability to obtain the proper particle morphology requires very careful control of temperature in the first step, and careful control of atmosphere in the second step. A two-step thermal process involving a rotary furnace followed by a pusher furnace resulted in production of a superior product. Significant experimental effort at the laboratory scale was necessary to identify the proper process conditions, and allowed for scaling to a successful production process.

The ultimate success of a process development activity can often be influenced by results and decisions made very early in the evaluation process. These decisions may later preclude certain equipment or process options that would otherwise make the difference in the quality, throughput or efficiencies of the commercial-scale product. The opportunity to evaluate new processes using commercially relevant equipment can assist in assuring that raw material selection, initial processing steps and assumptions regarding utilities consumption, product quality and process yield will meet the requirements of the market. When this testing is performed in conjunction with the commercial-scale equipment manufacturer, additional opportunities including obtaining equipment sizing data, matching process to equipment for maximised efficiency and producing sufficient material for customer testing are all gained. Ultimately, such a working arrangement maximises the opportunity for success, often in a reduced timeframe.

Further information: www.harperintl.com/resources

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