Performance Targets:

Achieving Operational Excellence in Chemical Manufacturing by Optimizing Contamination Controls

Entegris

INTRODUCTION

Operational excellence is of upmost importance when manufacturing high quality semiconductor grade chemicals and their raw materials. Streamlining and expanding operations, improving quality, and maintaining differentiation are all key activities toward achieving operational excellence. However, all these activities come at a cost; whether it be a financial impact or an impact on other opportunities.

Manufacturing personnel making raw materials and chemicals must focus on several metrics to provide a high quality product. Low levels of contaminants (particles and metals), acceptable health and safety profiles, and a streamlined manufacturing process are all requirements, which can all vary in importance to different customers.

This paper discusses the ways in which filtration and purification of raw materials and chemicals can improve quality by reducing metals and particles, improving purity, decreasing variability, and perhaps streamlining manufacturing processes.

CONTAMINATION CONTROL IN CHEMICAL MANUFACTURING

Chemical manufacturers have many considerations when identifying opportunities for contamination control. The entire value chain, from manufacturing to packaging, shipping, storage, installation, onsite mixing, and the fluid path to the wafer, influences an end user's measurement and quality perception, Figure 1. The chemical manufacturer has little influence on defectivity once the chemical enters the packaging. However, by preventing defectivity from ever entering the package, the manufacturer reduces the likelihood of a growing contamination problem.

While there are many places in the value chain that can introduce contamination, reducing contamination is possible during formulation and scale up. Those manufacturers who focus on reducing contamination have a competitive advantage, as they may shorten the customer's installation time while increasing their filter lifetime by lowering the total defect burden incoming to the system.

This competitive advantage, however, can come at a cost. Improving filter retention often comes at the cost of throughput because highly retentive filters may have higher pressure drops, resulting in lower flow rates. Adding more filters into the system is another option that can become costly and time consuming to manage.

While this may initially seem like a high barrier to overcome, there are several manageable solutions to improve purity during chemical manufacturing. The key is in collaboration.

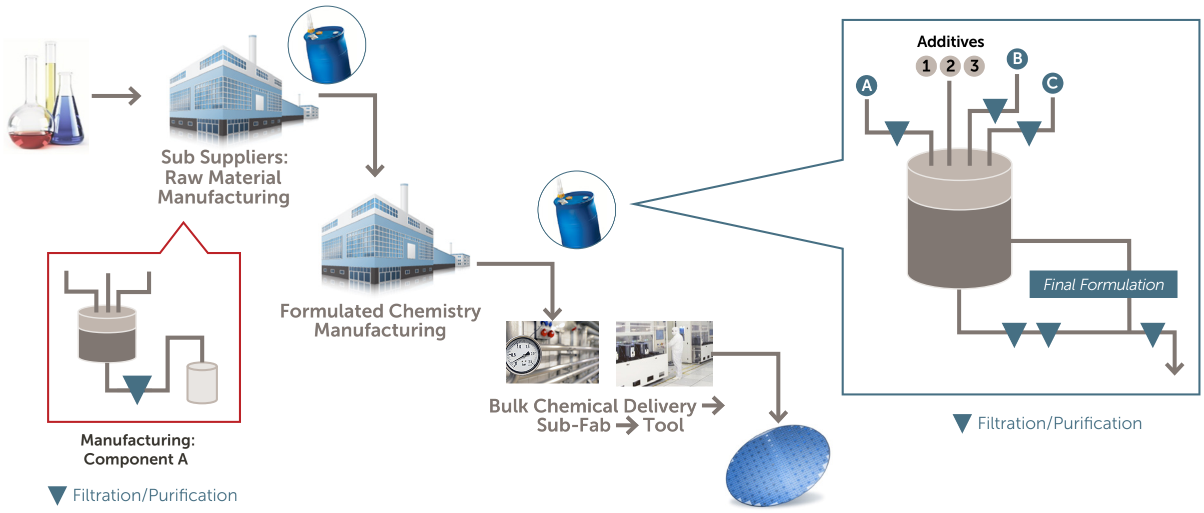

Figure 1. Specialty chemicals value chain. [1, 2]

PREDICTING THE OPTIMAL NUMBER OF FILTERS/PASSES FOR CONTAMINATION

In batch processes, chemistries are often recirculated to ensure the lowest contamination levels. But how many passes through a filter or purifier are enough? This can be hard to judge without investing in lengthy experiments. Entegris has modeled the retention behavior of different systems, large and small.[3]

To predict the behavior, the necessary inputs include:

- P&ID (piping and instrumentation diagram) of the system showing volumes of vessels, flows, and filtration points as a function of time
- Filter retention characteristics as a function of particle size and loading
- Particle size distribution in the source liquids

With this information, and some fundamental chemical engineering principles, it is possible to construct a mathematical representation of the system evolution as a function of time for any filtration point in the system.

Figure 2 shows the result of one model, comparing two different filters. Empircal data was collected using recirculation experimental setup where 105 L of water with a known concentration of polystyrene latex beads (PSL) was recirculated at five liters per minute. Samples were removed from a sample port at regular intervals to determine the filtration efficiency of different filters. Filter M achieved baseline more quickly than Filter L. This information can be used to optimize a filtration scheme to maximize benefits of upgraded filtration, including an optimized cost of ownership and an understanding of the reward or risk of upgrading filters.

Particle (PSL Beads) Removal vs. Tank Turnovers

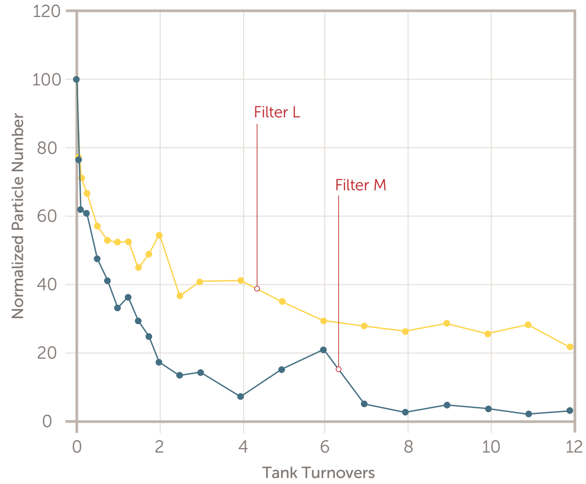

Figure 2. Filtration efficiency comparison. Filter M reaches baseline more quickly than Filter L.

A similar model has been created to understand the removal of metals from solvent systems. This model is more challenging because competing metal removal mechanisms in solvents are less understood. However, given an understanding of the filtration mechanism used in a particular system, a prediction can be made using adsorption kinetics and adsorption isotherms. In the experiment shown in Figure 3, Fe (iron) particles were introduced into an n-Butyl Acetate (nBA) recirculation tank. Different concentrations of Fe were measured prior to insertion to the system, and at regular intervals of tank turnovers using an Agilent 8900 ICP-MS (inductively coupled plasma mass spectrometer). From the model derived from this experiment, we can predict the number of turnovers required to eliminate a single metal from a solvent system. Further experiments and modeling efforts are ongoing to better understand multi-metal systems.[4]

Fe Removal % vs. Tank Turnovers

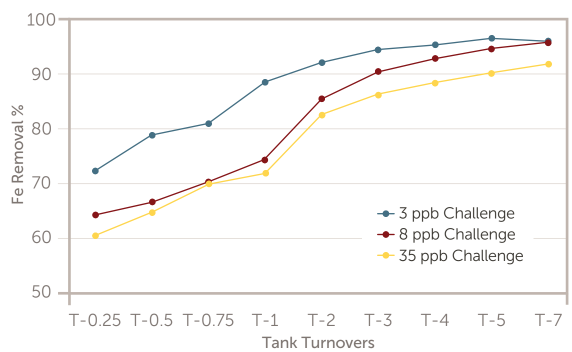

Figure 3. Removing different Fe concentrations from an nBA solvent system. Fe removal increases with increasing tank turnovers.

To support modeling efforts, Entegris provides opportunities to consult with field applications engineers, work with our laboratories on lab scale testing, and leverage our analytical capabilities for enhanced diagnostic testing. By collaborating with a filtration supplier, a chemical supplier can optimize the system before spending any time or money on experiments, thereby maximizing quality and manufacturing efficiency.

IMPROVING FLOW RATE WITHOUT CHANGING RETENTION

Identifying baseline particle counts by tank turnover is the first step to identifying improvement areas. Choosing a filter that achieves baseline quickly directly changes manufacturing efficiency without a significant change to the filtration scheme.

If you have already optimized flow rate and tank turns for your current filters, upgrading those filters to a higher flow solution at the same retention rating is the next step. This change will reduce the total number of filters needed, Figure 4. This change maintains contamination control and results in a smaller footprint for overall filtration, a reduction in the pressure delta across the system, and reduced filter disposal cost after use. A conservative chemical manufacturer could replace filters more often with this strategy to continue ensuring high quality chemicals, while benefiting from increased production efficiency.

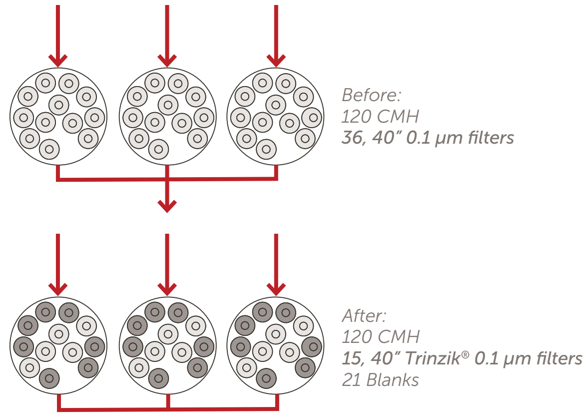

Figure 4. Before the filters were changed, this system used 36 total filters. After evaluating a higher-flow option at the same retention rating, the number of filters was reduced to 15.

BALANCED TRADEOFF BETWEEN CONTROLLING CONTAMINATION AND THROUGHPUT

Chemical manufacturers may also consider upgrading their filtration to a higher retention rating, maintaining their filtration footprint while upgrading in place. A more retentive filter will reduce overall defectivity but could create a higher pressure drop within a system, reducing throughput. However, some newly developed filters have been designed to have a less dramatic effect on throughput by maintaining lower pressure drop while increasing retention performance

In the case study presented in Figure 5, a filter was designed to be compatible with a specific chemistry. Initially, standard filters inserted into the filtration loop removed critical additives from the chemistry, rendering it less effective than predicted.[5] After studying the system as a whole, Entegris was able to design a better filter that would not interact with the additives, but would improve flow rate, retention, and particle loading performance.

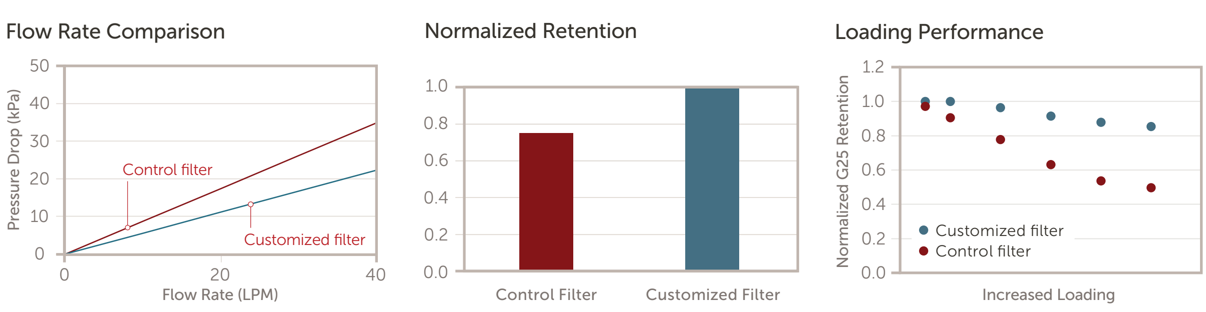

Figure 5. New filter designs optimize retention and flow rate to meet the needs of chemical manufacturers and end users alike.

CONCLUSION

Process changes are not often made in chemical manufacturing for fear of changing the end product, which may be shipped to specific control limits. However, there are opportunities to better meet customer needs, gain a competitive advantage, or improve throughput through filtration optimization. Filter suppliers have many tools at their disposal to predict filtration behavior and design filtration solutions to achieve operational excellence and high quality standards in chemical manufacturing facilities. Collaboration between chemical manufacturers and filter suppliers is critical for identifying the best opportunities to maintain or improve chemical manufacturing systems.

References

[1] *Reducing contamination points in clean chemical delivery from manufacture through point of use*, Entegris white paper, https://info.entegris.com/white-paper-reducing-contamination-points-in-clean-chemical-delivery-from-manufacture-to-point-of-use, August 2019.

[2] *Ensuring purity and safety: How to select the right container system for safe, clean chemical delivery*, Entegris white paper, https://info.entegris.com/white-paper-how-to-select-the-right-container-system-for-safe-clean-chemical-delivery, June 2019.

[3] Bhabhe, A., Ozzello, A., Qi, Y., Bradish, S., Seamster, K., *Empirical model to understand the particle removal behavior during bulk chemical filtration*, UltraPure Micro, (2018).

[4] Sampath, S., Bhabhe, A., Ozzello, A., Liu, L., Maharjan, K., Crepin, K., *Predictive model to understand removal of metals from solvents*, UltraPure Micro (2019).

[5] Bhabhe, A., Braggin, J., Phely-Bobin, T., *Contamination in Formulated Chemistries – How can filtration enable next generation semiconductor processes?*, UltraPure Micro (2019).

ABOUT ENTEGRIS

Entegris is a world-class supplier of advanced materials and process solutions for the semiconductor and other high-tech industries. Entegris is ISO 9001 certified and has manufacturing, customer service, and/or research facilities in the United States, Canada, China, France, Germany, Israel, Japan, Malaysia, Singapore, South Korea, and Taiwan. Additional information can be found at entegris.com.

FOR MORE INFORMATION

Please call your Regional Customer Service Center today to learn what Entegris can do for you.
Visit entegris.com and select the Contact Us link to find the customer service center nearest you.

CONTENT AND LIABILITY DISCLAIMER

Entegris

Corporate Headquarters
129 Concord Road
Billerica, MA 01821
USA

Customer Service
Tel +1 952 556 4181
Fax +1 952 556 8022
Toll Free 800 394 4083