



PROCESS SCALE-UP FROM DEMONSTRATION BATCH FILTRATION TO COMMERCIAL CONTINUOUS FILTRATION

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INTRODUCTION:

In the chemical process industry (CPI) as well as new technology arena (NTA), there are inherent risks and benefits to scaling up from the laboratory / bench top through pilot, demonstration and then finally commercial scale. This article will follow the process filtration approach developed for each technology stage gate starting from batch filtration in the lab to continuous filtration for the full-scale commercial operation.

The article begins with a discussion of each stage-gate and how to avoid the “cookie-cutter” approach to these types of projects. Process engineers must not “jump to conclusions”, a reference from Sherlock Holmes and Dr. John Watson who prove time and again is that there is no benefit to jumping to conclusions. Therefore, laboratory / bench top filtration testing is covered for problem analysis, technology selection and pilot and demonstration scale-up.

The paper continues with the filtration testing and then scale-up to the commercial size. Finally, the paper concludes with a general review of the problem-solving skills of Holmes and Watson and how these skills can be utilized by process engineers as a framework for “idea-generation” when analyzing a new process scale-up. In all cases, by combining Holmes and Watson with accurate filtration testing, the optimum filter selection can be realized at each stage.

TECHNOLOGY STAGE GATES:

Lab Scale

For the lab scale, the typical reactor sizing is 1 – 5 liters. These reactors can be simple batch precipitation reactions to more complex continuous-stirred tank reactors (CSTR). A frequent challenge at this stage is reliable solids and crystals size and shape. To address this, process researchers must test at different pressures, temperatures, times, agitation, etc.

Pilot Scale

Pilot plants provide the first view into the scale-up issues for the solids/crystals formation, feeding systems for slurries, handling and pumping issues, catalysts, etc. The volumes are larger up to 50 liters.

Demonstration Scale

Demonstration plants are different than pilot plants in that they are much closer to the commercial scale. They allow for more extended run times and typically produce the final product which can be checked for the various quality parameters. The capital costs are higher and there is more risk in this scale, generally up to 40,000 liters. This will be the go/no-go decision point for the commercial operation including investments, market information, internal and external forces, revenues, costs, etc.

Commercial Scale

The commercial scale can be of various sizes based upon the current and future market demand for the product. However, even if all of the previous work is optimized, the commercial scale can also develop problems based upon the assumptions made. In the book, Solid-Liquid Filtration, Practical Guides in Chemical Engineering, B. Perlmutter writes about interesting process challenges after the fact. There are small changes in the commercial scale that have large impacts on the process; think about Sherlock Holmes and identifying the crucial from the incidental.

CASE HISTORY: LAB-PILOT-DEMONSTRATION-COMMERCIAL SCALE-UP

According to Holmes and Watson, it is important to train yourself to be a better decision maker. For example, using checklists, formulas, structured procedures; those are your best bet.

LAB / BENCH TOP TESTING FOR PRESSURE AND VACUUM FILTRATION

The BHS bench top testing is conducted using the BHS Pocket Leaf Filter, as shown in Figure 1. The test device is a BHS pocket leaf filter with a filter area of 20 cm² and a vacuum and pressure connection. The testing will analyze cake depths, operating pressures, filter media, washing and drying efficiencies and qualitative cake discharge. The data collection sheets are shown in Figure 2. The steps in filtration testing are as follows:

First, it is necessary to clearly state the process description. This includes the slurry characteristics (particle size distribution, particle shape, density, etc.), washing of the cake (i.e. number of washes and wash ratios), drying / pre-drying of the cake (vacuum, pressure blowing, and mechanical pressing) as well as the upstream and downstream equipment. With this definition, the type of samples that need to be collected and analyzed can be determined.

Secondly, it is necessary to know what are the requirements for the operation such as solids/hour and cake quality (percent moisture, percent contaminants, etc.).

Thirdly, with the above in mind, the testing much determines the following objectives:

- Choice of a suitable filter cloth
- Vacuum or pressure filtration
- Wash ratios for the washing of the filter cake
- Drying techniques

For the initial testing for this project, the choice of the researchers was pressure filtration. The process had 1.8% solids and the requirement was batch filtration, cake washing, drying, solids to landfill and filtrate was sent to reactors for further processing. Due to the low solids content, the decision was made for candle filtration. Appendix A illustrates the candle filtration technology.

PILOT SCALE

Based upon the lab testing, the pilot stage installed two (2) BHS candle filters each with 0.2 m² of filter area. There are three batch reactors feeding the two candle filters. Figure 3 illustrates the pilot plant flow diagram. The pilot plant was planned for one-year operation.

After the pilot process began, the filter media did not produce the desired solids removal and cake discharge was difficult as displacement washing of the solids was not very efficient. The wash ratio was 10 kg liquid: 1 kg solid. Other issues surfaced including drying difficulties, cake cracking and variability of the cake thickness.

The key learnings at the pilot scale are as follows:

1. Vertical cake structure is hard to maintain
2. Drying beyond 80% moisture is not possible
3. High flux for filtration with little/no pressure buildup made it difficult to determine the end of filtration
4. Wash ratio of 10:1 generates acceptable product yield but not efficient
5. Tight filter cloth was needed for filtrate quality

In summary, the candle filter was not the optimal technology. This was an important discovery at the earlier stages of the project. The decision was to go back to the lab testing to optimize the filtration process for the demonstration scale plant.

According to Holmes and Watson, it is easy to succumb to certainty but every time you find yourself making a judgment upon observation, train yourself to stop and repeat. Then go back and restate from the beginning and in a different fashion and most importantly, out loud instead of silently, as this will save you from many errors in perception. Process engineers can benefit from discussing options with technology suppliers that can provide different filtration solutions.

LAB SCALE TESTING FOR DEMONSTRATION SCALE PLANT

For this lab testing, the focus was on vacuum filtration based upon the pilot plant experience. Horizontal continuous – indexing vacuum filtration is beneficial for cakes with:

1. Very little cake resistance
2. Horizontal cake structure
3. Good for multiple washes
4. Mechanical pressing could yield improved cake moisture

The results of the lab testing are as follows:

1. Solids concentration was increased to 7% from 1.8% which improves the process and allows for vacuum filtration
2. Filter media, 7 micron (um) produced clear filtrate, non-detectable fines
3. Cake thickness is reproducible, 50 mm, and cake discharge is optimum, see Figure 4
4. Filtration cycle time is 25 seconds
5. Cake washing is 20 seconds
6. Displacement washing with water is very efficient to > 90%
7. Neutralization is very efficient for 70 seconds with reduced caustic concentrations
8. Vacuum drying in combination with pressing and blowing for 50 seconds yielded consistent dryness > 80% and a reduction in cake thickness to 30 mm for easily solids handling for disposal.

In summary, for the demonstration scale plant, the decision was made for a continuous-indexing vacuum belt filter. Appendix B illustrates the technology.

DEMONSTRATION SCALE PLANT

For the demonstration plant, based upon the lab testing, one continuous-indexing vacuum belt filter was installed of 0.6 m² in filter area including vacuum pumps and vacuum separators with liquid transfer pumps. Figures 5 and 6 show the vacuum filter and vacuum pump – filtrate skid. The vacuum filter has twelve (12) zones to allow for filtration, cake washing, neutralization, and drying with vacuum, mechanical pressing and blowing.

The demonstration plant has completed its operation and the laboratory testing was validated for the commercial scale operation.

COMMERCIAL SCALE PLANT

The commercial plant design is shown in Figure 7. It consists of eleven (11) stages of operation for filtration, cake washing, neutralization and drying. The process guarantees are based upon the demonstration plant for production rate, wash ratios and quality and final dryness. The process is now waiting for final capital approval.

CONCLUDING REMARKS & TAKEAWAYS:

It is important to consider all of the steps in process scale-up. First, it is critical to obtain the correct data from all perspectives including reaction, filtration, solids handling, drying as well as all of the other upstream and downstream equipment and systems. The team must know the process, observe the testing, and deduce the solution only from what is observed (and nothing more). Partnering with suppliers with a proven track record in similar applications will shorten the technology scale-up cycle. Secondly, always allow time for fine-tuning even after the scale-up seems complete. Next, the start-up and commissioning at each step will also have unknowns associated with these activities.

Finally, throughout this article, there are references to Holmes and Watson and their unique view of problem solving. The world of a process engineer is a distracting place and Holmes and Watson know that without the occasional silence, as in *The Hound of the Baskervilles*, there can be little hope for success. The team requires time to think about the process issues, at hand, before moving into the piloting and demonstration stages. Holmes and Watson excel at “deduction from facts and deduction difficulties.” All that matters is what the premises are (process definition, requirements and testing objectives) and how the testing unwinds the crucial from the incidental (what is the critical process parameter) and finally ending up in the logical conclusion (optimum process filtration solution).

It is always difficult to apply Holmes’ logic but as Holmes’ states “you know my methods, now apply them.” Engineers must practice these habits such that even under stress of a scale-up, these stressors will bring out the very best thought patterns that are needed.

References:

1. All information about Holmes and Watson is taken from Maria Konnikova's *Mastermind-How to Think Like Sherlock Holmes* (Viking Penguin (USA) Inc. 2013).
2. All information about solid-liquid filtration is taken from Barry A. Perlmutter's *Solid-Liquid Filtration, Practical Guides in Chemical Engineering* (Elsevier Inc. 2015).
3. All general information about scale-up ideas are based upon BHS experience and the following articles: *Scaling-Up Bioenergy Technologies*, David Edwards (Chemical Engineering Progress, March 2015, pages 58 – 61) and *Inside Look at Financing Biorenewable Projects*, Ron Cascone (Hydrocarbon Processing, April 2015, page 21).
4. All information about this project is based upon BHS process testing and development with a confidential client.

Figure 1: BHS Pocket Leaf Filter

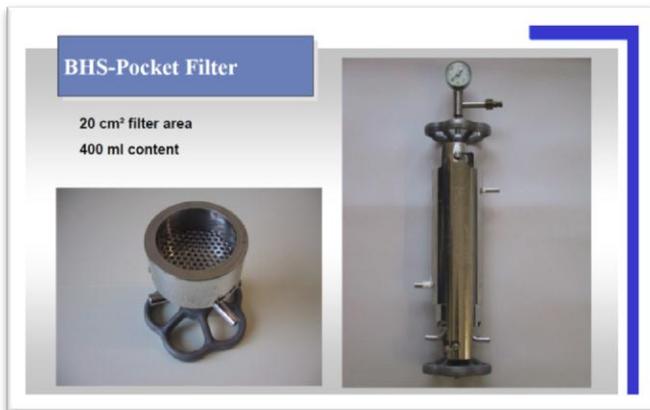
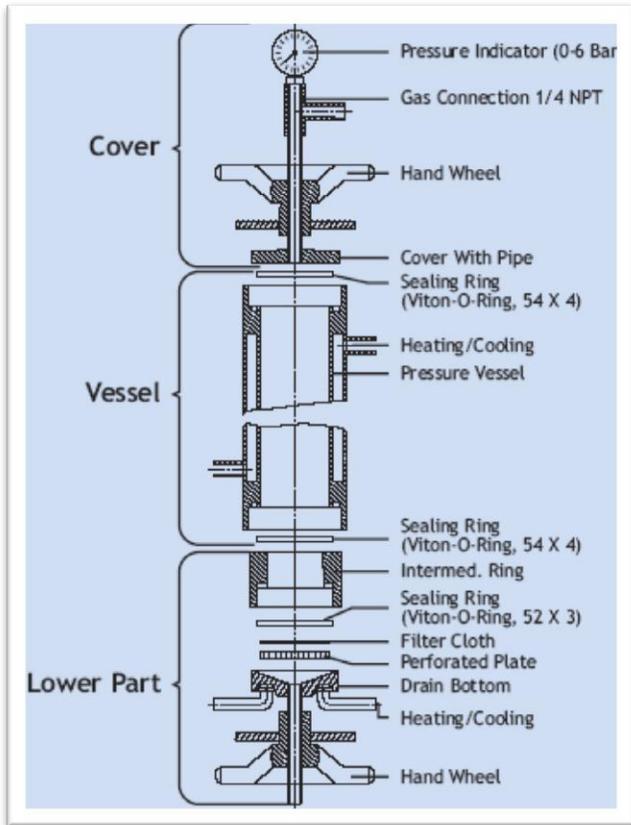


Figure 2: Data Collection Sheet for BHS Pocket Leaf Filter

Customer:	
Date :	
	Filter Media Suspension
Filling	Volume of Slurry Density of Slurry % Solids in Feed Temperature
Filtration	Pressure/Vacuum Volume of Filtrate Time for Filtration % Solids in Filtrate
Wash 1	Wash Material Pressure/Vacuum Volume of Filtrate Time for Filtration
Wash 2	Wash Material Pressure/Vacuum Volume of Filtrate Time for Filtration
Wash 3	Wash Material Pressure/Vacuum Volume of Filtrate Time for Filtration
Drying	Pressure/Vacuum Temperature Flow Rate Time for Drying Pressing Pressure
Cake	Weight Thickness % Residual Moisture Dry Cake Weight Cake Discharge OK?

Figure 3: Process Flow Diagram

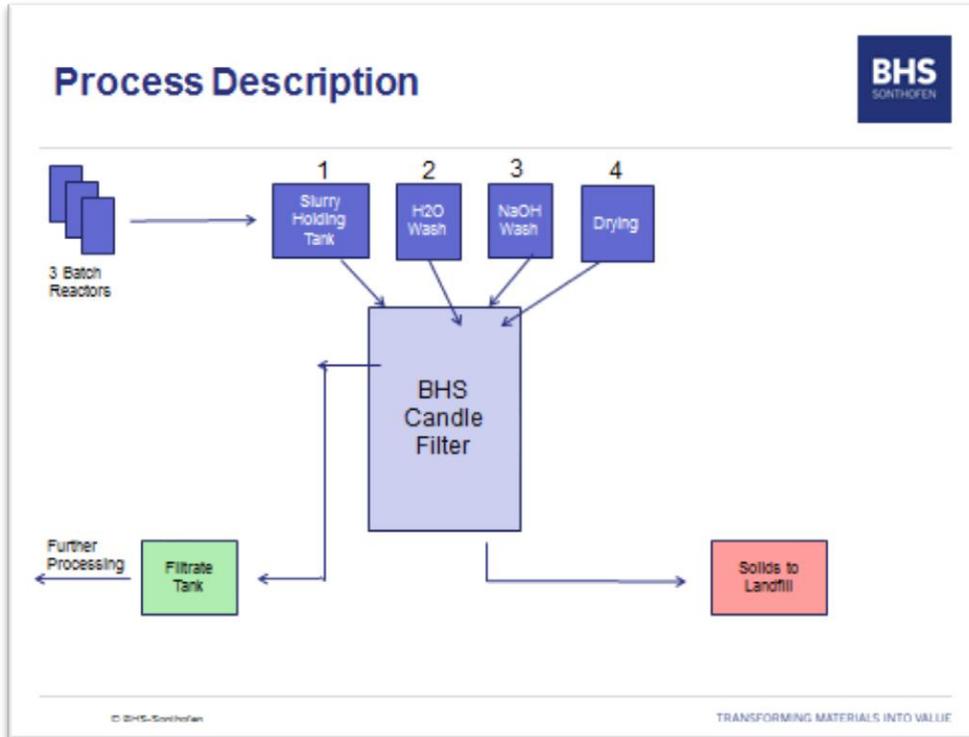


Figure 4: Cake Structure and Cake Discharge from Vacuum Filtration



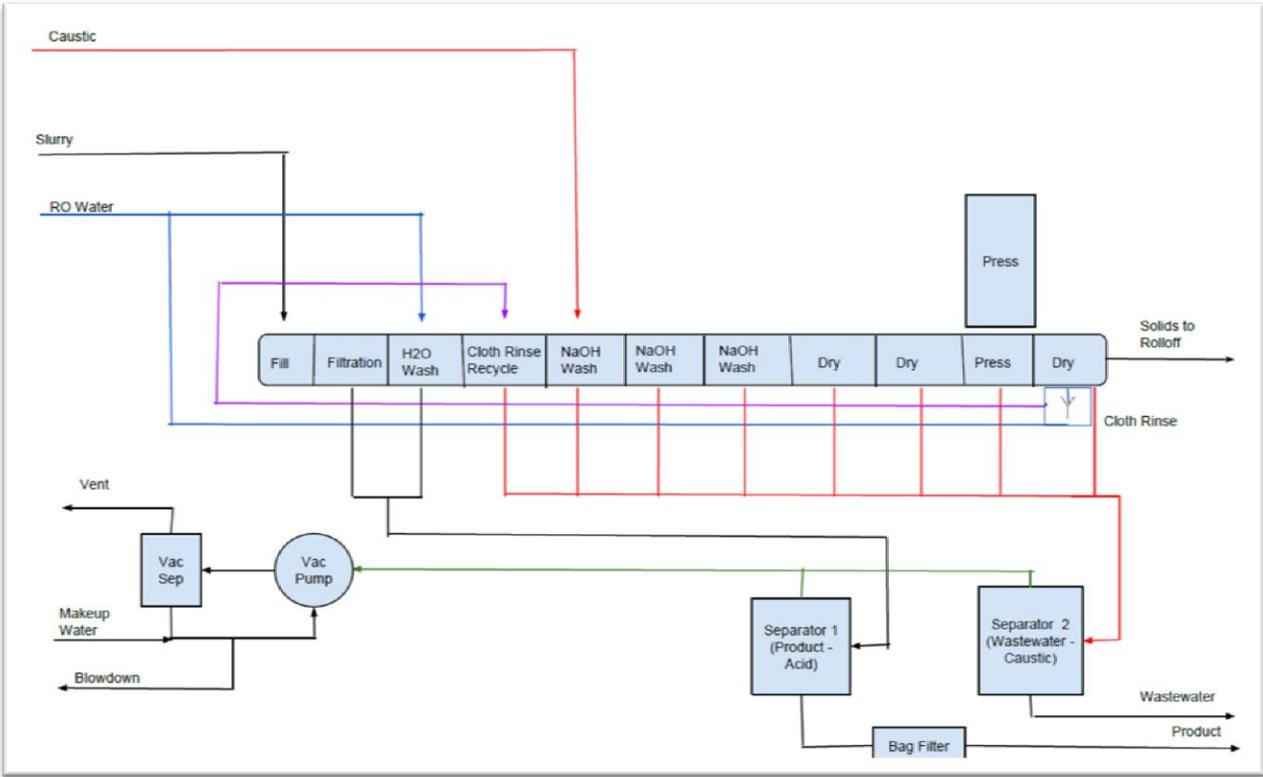
Figure 5: BHS Continuous-Indexing Vacuum Belt Filter, BF 025-024 (0.6 m2 filter area)



Figure 6: BHS Vacuum-Filtrate Skid for BF 025-024 (0.6 m2 filter area)



Figure 7: Commercial Scale Design



APPENDIX A: CANDLE FILTRATION TECHNOLOGY

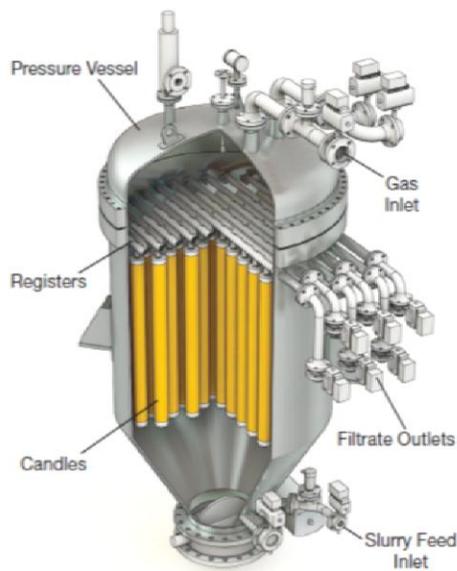
A candle filter is a pressure vessel filled with tubular filters called filter candles (Figure 1). A typical filter candle is comprised of a pipe to conduct the filtrate and pressurized gas, a perforated core with supporting tie rods, and a filter sock (Figure 2). The filtrate pipe runs the length of the candle and ensures high liquid flow, as well as maximum distribution of the gas during cake discharge. The tie rods create an annular space between the filter sock and the perforated core, which helps to maintain a low pressure drop during operation and promotes efficient expansion of the filter sock during cake discharge. The filter sock is installed over the candle, and can be made of various synthetic materials, capable of removing particles smaller than 1 μm . As the cake builds during operation, the candle filter's removal efficiency increases, enabling removal of particles as small as approximately 0.5 μm .

The candles are installed in a pressure vessel constructed of stainless steel or another alloy. Within the vessel are horizontal manifolds called candle registers. Each candle is connected to a register with a positive seal to prevent bypass. Depending on the filter size, each register may contain 1–20 candles. The liquid filtrate and pressurized gas flow through the register; automated valves ensure optimum flow in both directions.

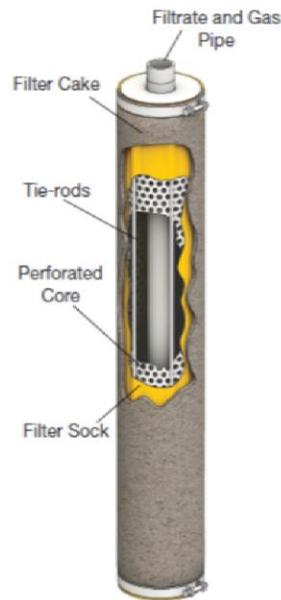
During operation, a feed pump or pressure from the reactor or feed tank forces the slurry into the bottom of the pressure vessel. The solids build up on the outside of the filter sock, while the liquid filtrate flows into the candle, through the registers, and out of the vessel. This process continues until the maximum pressure drop, design cake thickness, minimum flow, or minimum filtration time is reached.

The cake is washed to remove impurities and leftover mother liquor, and then dried. Next, low-pressure gas enters the individual candles and expands the filter socks. This process breaks apart the dry cake, which detaches from the filter sock (Figure 3) and falls into the vessel cone. The cake can also be discharged as a concentrated slurry.

Candle filters are used for thin-cake (5–20 mm) pressure filtration applications. They are best suited for filter cakes that are stable vertically because of the orientation of the candles.



▲ **Figure 1.** In a candle filter, the slurry enters through the bottom of a pressure vessel and flows across the filter media. The filter candles are attached to registers that collect the filtrate. Gas is fed into the top of the pressure vessel for cake drying and discharge.



▲ **Figure 2.** During operation, filtrate exits from the top of the candle, while the solids collect on the synthetic filter sock.



▲ **Figure 3.** During discharge, gas is fed into the top of the candle, which expands the flexible filter sock. This causes the dry cake to crack and break away from the filter. The solids are collected at the bottom of the pressure vessel.

APPENDIX B: CONTINUOUS-INDEXING VACUUM BELT FILTER TECHNOLOGY

The Continuous – Indexing Vacuum Belt Filter (CI-VBF) consists of fixed vacuum trays, continuously feeding slurry system and indexing or step-wise movement of the filter media. Figure 1 illustrates, in practical terms, the operational features of the CI-VBF, which, from a testing point of view, can be a series of Buchner funnels.

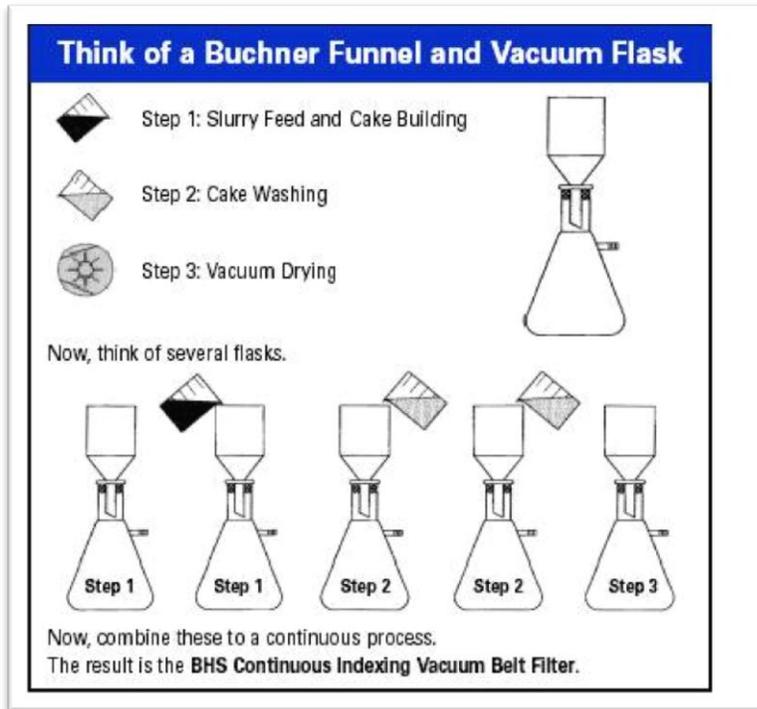


Figure 1: Buchner Funnels Illustrating the Continuous-Indexing Vacuum Belt Filter

In the CI-VBF, the slurry feed is continuous while the filter cloth is moved intermittently; the trays are fixed in place. For cloth movement, the vacuum is broken by butterfly valves and the cloth moves (indexes), by an electric motor, in the space above the vacuum trays. The belt moves along and relaxes while indexing, which allows for long filter cloth life. The CI-VBF eliminates the need for rubber carrier belts to move the filter media. Further, compared with moving tray designs, the CI-VBF requires no additional hardware such as rails, rollers and flexible pressure vacuum rated hoses that are within the belt filter frame.

For the process operation, due to the stepwise operation of the belt, washing and drying efficiencies are maximized with the stopped belt and the mechanism of “plug-flow” for gases and liquids is in effect. Finally, the fixed trays allow for the mother liquid and the wash filtrates to be recovered individually and recirculated/recovered/reused for a more efficient operation.

After filtration, washing and drying, the cake is discharged and the filter media is cleaned. Cleaning occurs only when the filter belt is moving which ensures maximum cleaning with lower liquid usage. Automated valves control the washing. The clean filter media is then conveyed back to the slurry feed zone.

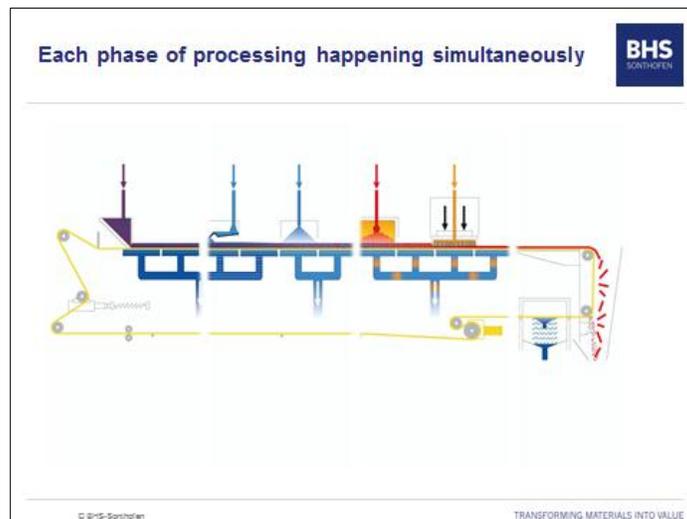
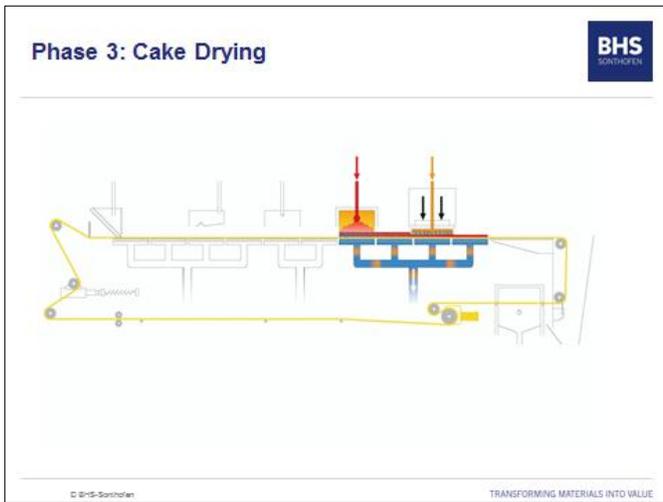
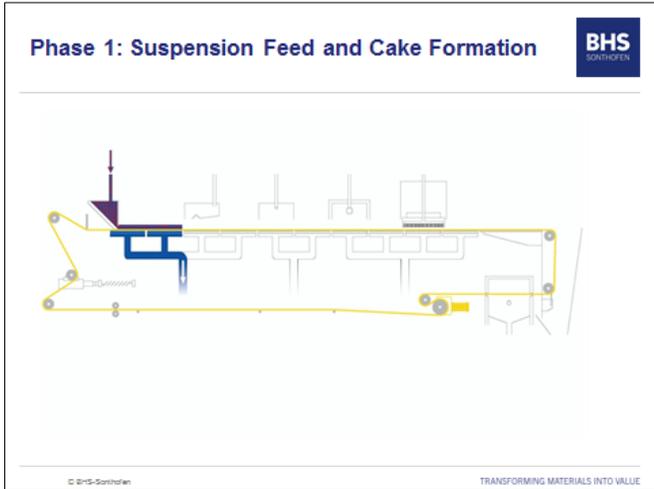


Figure 2: Schematic of the Continuous-Indexing Vacuum Belt Filter