A Six Sigma Approach to Evaluating Vacuum Filtration Technologies

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The appropriate testing procedures help direct the selection of vacuum filtration equipment and ensure optimum equipment operation.

Solid-liquid separation can be accomplished by pressure or vacuum filtration in a batch or continuous mode. Generally, following filtration, the cake must be washed and subsequently dried to specifications. The technology choice process depends on a wide range of parameters, including the nature the solids (crystal size and distribution, shape and type), the liquids (temperature, viscosity and nature) and the process itself. This article examines the use of a continuous-indexing vacuum belt filter (CI-VBF) as a replacement for manually operated filter presses and offers insight into one plant's technology-selection process.

A specialty chemical plant was using several filter presses to produce various high-valued products in the form of thin filter cakes. Each unit contained 136 plates with a total filter area of 4,400 ft^2 (440 m^2). Unfortunately, the plant was experiencing a wide range of operating and process problems:

• excessive use of wash liquid and requirement of high wash ratios to meet a conductivity specification

• high moisture content in the discharged cake required longer drying times

• frequent filter-media blinding resulted in high maintenance costs for changeouts

• manual processes, such as discharge of cake from the filter press, raised operating costs

• difficulty in handling the wet-cake discharge required frequent operator intervention

• mixing of the mother liquor and wash filtrates increased the costs of distillation and liquid recovery.

STEP 1. Establish Performance Criteria and Pick Technology with Highest Marks

The plant engineers took a Six-Sigma approach to improving the filtration process that involved replacing the existing equipment with centrifuges, nutsche filters or vacuum belt filters (VBF; moving tray, rubber belt and continuous-indexing designs). The criteria for the initial evaluation included continuous operation, multiple washing stages in the forward and countercurrent modes, ease of cleaning, cake compression, thermal drying features to recover drier solids and to minimize the quantity of drying equipment (*i.e.*, reduced capital expenditures for the project).

A primary consideration was the high compressibility of the product cake. On a nutsche filter-dryer, this cake would necessitate long cycle times and potentially cause cloth blinding. The engineers suspected that centrifuges would have similar cycle-time and blinding issues. Another crucial requirement was thorough washing of the cake. Tests revealed that centrifuges and filter-dryers did not satisfy this criterion. Since the VBF met the first two objectives and also operated continuously, it was designated as the preferred technology. The engineers then compared the three available VBF technologies to find the one that was most suitable for their application. Table 1 illustrates this decision-making process, which led to the selection of the CI-VBF system for further bench-top and pilot-scale testing.

Figure 1 depicts the operational features of the CI-VBF, while Figure 2 illustrates the process technology. The slurry is fed continuously from above and spreads out evenly onto an indexing (or intermittently moving) horizontal belt. In other words, the cloth is moved during the feeding of the filter and stopped during the suspension dewatering obtained by the vacuum. The filtrate drains through the belt while the cake is carried along the belt and discharged at the end. Belt speed and drying time depend on the desired characteristics of the cake and can be modified to obtain the best results.

How the CI-VBF operates

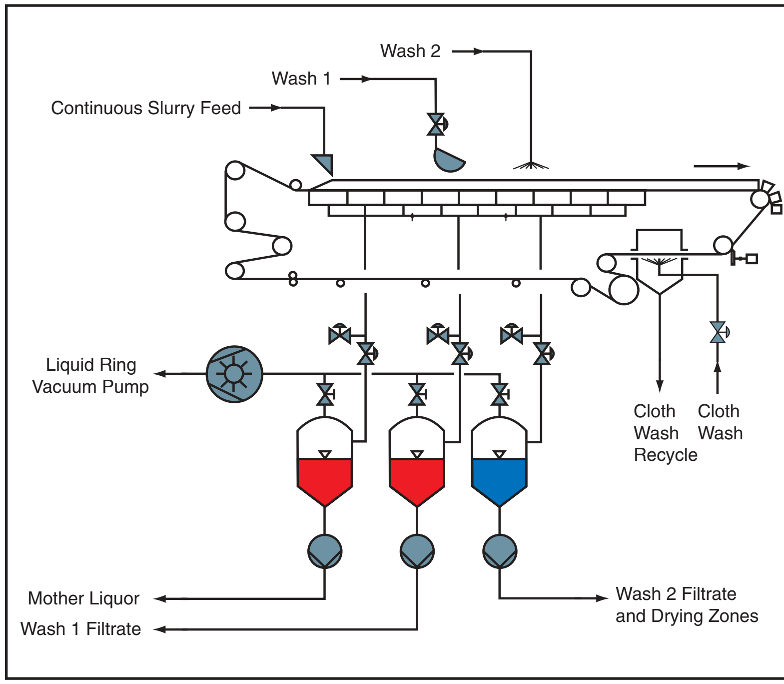
Vacuum tray design. Constructed of synthetic material, stainless steel, Hastelloy or similar metals, the vacuum trays are located under the filter belt. For indexing filters, they are divided up into segments, or zones, each of which is connected separately to the filtrate manifold. Because the trays are fixed in place, the mother liquid and the wash filtrates can be recovered individually and recirculated and/or recovered and reused, which allows for a more efficient overall operation. The filter belt never touches the trays, which allows for long filter belt life.

Cloth movement. The filter cloth moves intermittently at a variable belt speed. Movement occurs when the vacuum on the filtrate pipe is released by the rapid opening and closing of butterfly valves (also called shut-off valves) as the belt is being conveyed.

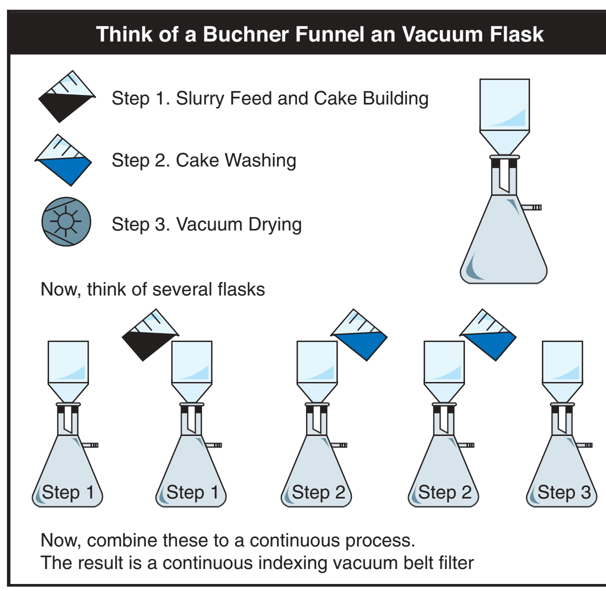
Belt mechanisms. The CI-VBF eliminates the need for rubber carrier belts and a motor to move the filter media. In contrast to other moving tray designs, it does not require additional hardware, such as rails, rollers and flexible pressure-vacuum rated hoses, which reside within the belt filter frame. For intermittent belt filters, the extraction roller pulls the belt forward, while the locking roller restrains the lower belt section. The conveyed cloth length is then extracted from the belt storage device. Once the extraction roller returns to its home position, the locking roller is released and the belt storage device takes the filter cloth back up again.

Two opposing roller pairs keep the filter belt on course. If the belt moves from its preset path, a belt-edge sensor gives a signal to slacken the roller tension on the side in distress. The second roller pair returns the belt to its intended position.

Washing of the cake. The filter cake undergoes one or more stages of dilution or displacement washing when the belt is stopped to purge contaminants that are suspended or dissolved in its its pores. When the belt is stopped, the mechanism of "plug-flow" for gases and liquids is in effect, maximizing washing and drying efficiencies. The washing liquids and washing filtrates from countercurrent washing, as well as the different liquid media for extraction or ion-exchange processes, are fed to the filter co-currently or countercurrently through distribution and feed channels or spray nozzles. In addition, when the belt is stopped, the filter cake may be washed in a particular zone. If necessary, the vacuum process can be delayed to allow the washing media to penetrate the filter cake.



■ Figure 1. Process flow diagram of a continuous-indexing vacuum belt filter.



■ Figure 2. Modified stage-gate framework for process development.

Delivery and recovery of wash liquids. After the washing liquid displaces the residual mother filtrate from the cake, the resultant wash filtrate flows through the manifold for disposal or further processing. As required by the process, the mother and wash filtrates may be combined or delivered to separate tanks.

Dewatering and drying the filter cake. Following the normal filtration and washing stages, various dewatering systems may be used to dry the product. When the filter

Table 1. Comparisons of vacuum-belt filter (VBF) technologies.

Component	Continuous Indexing (CI-VBF)	Moving Tray	Rubber Belt	Comments
Rubber Belt and Sealing Belts	Not required	Not required	Required; movement is via an external motor	Increased maintenance and spare-part costs; increased water usage (30%) for belt sealing and lubrication
Moving Trays	Not required	Required	Not required	Rails and rollers must be maintained/lubricated; flexible hoses for the filtrate outlets are difficult to reach and may require an enclosed space entry
Slurry Feeding	Gentle; eliminates splashing; oscillating feed zone is possible	Inclined feed plate	Inclined feed plate	CI-VBF employs a controlled feed for complete tray coverage and even cake buildup
Cake Washing	Efficient displacement wash as belt is stopped; residence time can be maximized	Continuous	Continuous	CI-VBF employs spray nozzles or overflow for washing; cake washing is controlled and efficiency maximized
Multiple Stages of Cake Washing	Yes	Yes	Yes	Possible with all designs
Separation of Mother Liquors and Wash Filtrates	Yes; trays and filtrate outlets are fixed so each filtrate can be recovered separately	No; filtrates are mixed making recovery difficult	No; filtrates are mixed making recovery difficult	CI-VBF allows for recovery of the mother liquor and filtrates; recirculation and reuse is easily accomplished
Mechanical Cake Compression	Yes, belt is stopped	Yes, design requires a squeezing belt against the discharge roller which also rubs the cake	No	CI-VBF allows for mechanical squeezing of the cake
Drying by Vacuum	Yes	Yes	Yes	In some cases, vacuum levels may be higher in CI-VBF
Drying by Hot Gas Blowing or Steaming	Yes; belt is stopped which allows for steam or gas to be directly injected into the cake; can be used with open designs	Yes, heats up air space, but not the cake directly; requires a hood	Yes, heats up air space, but not the cake directly; requires a hood	CI-VBF provides for maximum drying efficiency in open or pressure-tight designs
Filter Belt Cleaning	Yes	Yes	Yes	CI-VBF units can employ different methods of belt cleaning, including from above and below the belt, high-pressure spray nozzles; wash water is only used when the belt is moving; the wash water is stopped when the belt is stopped, which reduces water and/or solvent usage
VBF Cleaning	Easy to clean	Difficult	Difficult	CI-VBF units eliminate extra hardware and belt components for easy cleaning; automated clean-in-place designs without operator intervention are possible

cake leaves the first wash zone, it is saturated with liquid. During the dewatering process, which starts when the cake is completely saturated, a vacuum is applied to overcome the capillary pressures in the cake. Drying air flows through the pores as they open up and reduces moisture.

Discharging the filter cake. Indexing belt filters discharge part of the filter cake during forward feed over the deflection roller at the back end of the belt filter. The remainder is deposited into the discharge hopper as the extraction roller returns to its home position.

Filter cleaning takes place in the downstream rinsing chamber and is conducted only when the belt is in motion. Typically, the cloth is sprayed intensively on the cake side, the reverse side or on both sides and, if necessary, dried by the vacuum system. This maximizes the filter cloth life and permeability to ensure efficient belt filter operation.

CI-VBF units can be enclosed, dust-tight, or pressure tight for inerting or gas-blanket and are manufactured in stainless steel, Hastelloy, synthetic or reinforced-synthetic components, depending upon the solvents, solids, temperatures, etc. The entire operation is pneumatic and controlled by a programmable logic controller (PLC) or distributed control system (DCS), which minimizes the installation, maintenance, water batching and process startup time.

STEP 2. Conduct Laboratory Tests with a Pocket-Leaf Filter

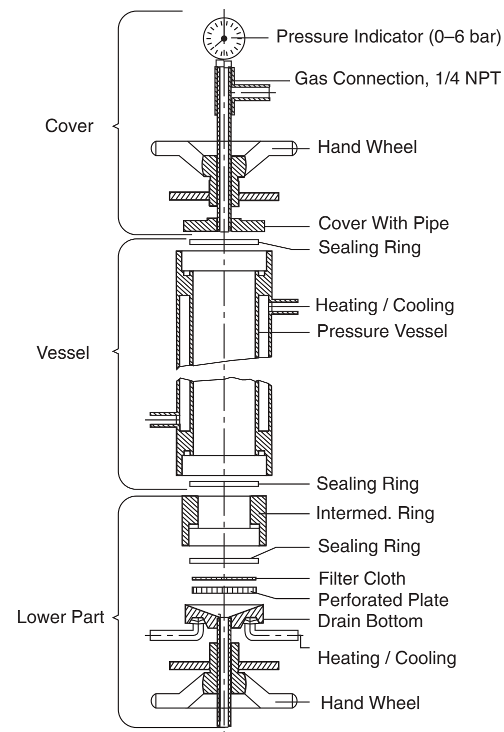
Bench-top testing narrows the gap between theory and practice and initiates the equipment design process. One useful bench-top filter system is the pressurized pocket-leaf filter (PLF), which resembles a Buchner funnel (Figure 3). In the project discussed here, preliminary bench-top tests were conducted in the vendor's laboratory using a PLF with 20 cm^2 of filtration area. A number of items are required for accurate PLF testing, including:

- material safety data sheets (MSDS) for all materials
- 4,000–8,000 mL of representative feed material for each sample to be tested
- 2,000 mL of wash material for each wash
- a 1,000–4,000-mL closed container with a mixer to prepare the feed material before each run
- several 250–500 mL containers for the feed material, the filtrate, the fresh wash material and the wash filtrates
- small containers for the filter cake
- a scale that measures gram quantities
- a vacuum oven or other instrument/equipment to check the percent solids in the feed slurry, filtrate (mother liquor) and wash filtrates, as well as the percent moisture in the filter cake via a Karl-Fischer analysis or an alternative technique.
- gloves and breathing equipment
- a regulated air or gas supply that can be controlled at 90 psig
- a flowmeter on the air or gas supply to allow the flowrate to be measured during the drying step
- a heat-transfer medium (hot oil, steam or cooling liquid)
- a vacuum source
- a specific test apparatus to measure data such as conductivity, particle size after completion of the testing cycle, etc.

PLF testing procedures and results

1. Pressure or vacuum filtration. The objective of this test is to minimize the vacuum filtration cycle time and reduce and/or eliminate the amount of solids (fines) lost in the filtrate. A pre-measured amount of slurry is added from the top of the apparatus, vacuum is applied, and the amount of filtrate vs. time is recorded.

For thin-cake filtration technologies, cake depths can vary from 5 to 25 mm. Maximum cake thickness for the PLF unit is 150 mm. The vacuum filtration tests performed in this case showed that a 8–10-mm thick cake produced with a 7-μm (pore size) filter medium meets the objectives.



■ Figure 3. The PLF consists of a pressure vessel (90 psig to full vacuum), a top cover with a pressure gauge and gas (or air) connection and a base for the filter media and filtrate outlet. Materials of construction include 316 Ti stainless steel, Hastelloy or polypropylene, and the fill volumes range from 250 to 2,000 mL.

2. Displacement washing. The PLF simulates the plug-flow displacement washing that occurs in the CI-VBF. This procedure is performed after the filtration step and is designed to minimize the wash ratios while consistently meeting the conductivity specification. A measured amount of wash liquid is added at a predetermined wash ratio (without disturbing the cake) and pressure and time are measured. One or more wash tests may be conducted with the same or different wash liquids. The engineers examined the impact of temperature along with forward and countercurrent washing. Forward-flow washing at approximately 60°C was found to be the optimum process condition.

3. Cake pressing. The CI-VBF may be designed with a mechanical device that compresses the cake when the tray stops moving. The PLF simulates this mechanism with a "pressing plug" that is actuated by pressurized nitrogen during the drying step and squeezes the cake onto the filter media. Due to the compressibility of the cake in the current study, compression was not successful.

4. Drying. Product drying in the PLF is tested by blowing ambient-temperature or hot gas through the cake or, as in this case, by creating a vacuum. Both the vessel jacket and base jacket are heated to simulate a production unit. The vacuum is kept constant, and gas throughput is measured vs. time. After a preset drying time, the cake is removed, and its depth,

Table 2. Data collection form for pilot-scale test.

Customer: ____________ Report #: _________

Date: ________________ Test Unit: _________

		Run #1	Run #2	Run #3
Set-Up Information				
	Indexing Time, s			
	Stopped Time, s			
	Indexing Length, mm			
	Vacuum, mbar			
	Suspension Name			
	Density of Slurry			
Feeding	Feed Rate of Slurry			
	Temperature of Feed			
	Number of Filtration Zones			
	% Solids in Filtrate			
Wash 1	Wash Material			
	Flowmeter Reading of Wash			
	Actual Wash Flowrate			
	Temperature			
	Number of Wash 1 Zones			
Wash 2	Wash Material			
	Flowmeter Reading of Wash			
	Actual Wash Flow Rate			
	Temperature			
	Number of Wash 2 Zones			
Total Wash	Total Wash Ratio in L wash/L feed			
Drying	Pressure on Pressing Device			
	Temperature			
	Number of Drying Zones			
Cake	Wet Cake Mass Flowrate			
	Dry Cake Mass Flowrate			
	Residual Moisture, %			
	Thickness, mm			
	Cake Quality			

weight and moisture content are measured. Several iterations are required to determine the optimum time and final moisture content. In this case, the cake was easily removed from the filter media with no residual heel.

5. Results and analysis. After completing these tests, the process engineers analyze the data and write a report. This document includes an executive summary, test objectives, test methods and facilities, test data (table format), test results (in written and graphical form, including filtration and drying curves), recommendations of production equipment and scale-up procedures and any other "path-forward" steps.

Based on the PLF tests and recommendations, pilot-scale tests are conducted. It is best to perform these experiments at the plant site using actual feed material from the reactor, as well as the actual washing and drying media, operating conditions, etc. It is crucial that the engineer who conducted the PLF tests also conducts the pilot tests. This will ensure that the procedures and testing "tricks" employed at the bench-scale will be preserved.

STEP 3. Calculate Scale-Up Parameters

The vendor and the customer's Six-Sigma team selected the most representative test results for the scaleup calculations. The scale-up procedure comprises a set of equations in which: F is the filter capacity, L/m^{2}min; V is the sample volume, L; P is the production rate, L/min; t_f, t_w and t_d are the filtration, total washing and total drying times, respectively, min; A_{PLF} is the area of the pocket-leaf filter; A_f is the required filtration area for the given production rate; A_w and A_d are the washing and drying areas, respectively; and A_t is the total filtration area required to meet the new production rate (area is in m^2).

Using the laboratory-scale values of V, A_t and t_f, calculate F.

$$F = V / (A_{PLF} \times t_f) \quad (1)$$

Use F, P and the laboratory-scale values of t_f, t_w and t_d to calculate A_t for the pilot-scale system:

$$A_t = A_f + A_w + A_d \quad (2)$$

$$A_f = P / F \quad (3)$$

$$A_w = (t_w / t_f) \times A_f \quad (4)$$

$$A_d = (t_d / t_f) \times A_f \quad (5)$$

STEP 4. Perform Pilot Tests at Customer's Site

Preliminary tests and scaleup calculations indicated that the product could be recovered by vacuum filtration, washed with a 20% reduction in the wash ratios (while meeting the conductivity requirements) and dried to a desirable cake. The belt filter area required to meet the production rate was computed as 18 m^2. Pilot-scale testing proceeded at the customer's site to: confirm these filtration, washing and drying results; establish the required size of the CI-VBF; discover the tendencies of the cake to form cracks or exhibit processing or handling-variability issues; and allow the operators to see the CI-VBF in operation.

The design of the pilot-scale CI-VBF is based on process parameters, the material of construction of the belt and other considerations. In the present study, two test units were offered — a 0.3-m^2 turnkey unit made of stainless steel and contained in a pressurized housing for gas inerting and a 0.8-m^2 turnkey unit made entirely of polypropylene and sporting an open design. They were equipped with a liquid ring vacuum pump, filtrate receiver tanks, liquid transfer and recirculation pumps, instrumentation, and a pneumatically driven control system, which, for pilot tests, allows for easy installation without concern for the building's electrical classification

Since containment was not required, the team opted for the 0.8 m^2 design. Table 2 illustrates the data collection form used in the pilot tests. Scaleup calculations for commercial production were performed in accordance with Eqs. 1–5. The computed value of A_t was then increased by adding in the area of the feed and discharge trays. From these pilot scale tests, it was determined that an 18-m^2 CI-VBF, including a built-in safety factor for increased production, would meet the current and future production needs of the plant.

STEP 5. Install a Pilot CI-VBF System

The CI-VBF is currently producing validated product and meeting the production objectives of the plant. The time from delivery through installation, mechanical commissioning, water-batching and process startup was approximately three weeks. Compared with the 440-m^2 filter press, the CI-VBF is providing improved washing efficiency for a higher quality product, and significantly lower operating and maintenance costs. The return-on-investment was realized in 3-6 months. CEP

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