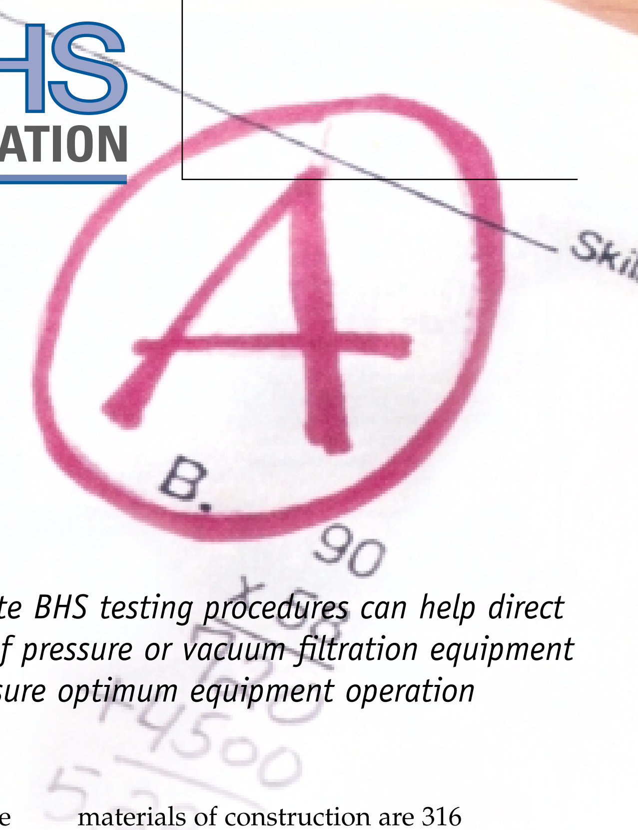
BHS FILTRATION



Test it Right

For Thin-Cake Filtration Operations

The appropriate BHS testing procedures can help direct the selection of pressure or vacuum filtration equipment and ensure optimum equipment operation

By Barry A. Perlmutter

Solid-liquid separation by pressure or vacuum, cake washing and drying are integral to producing a chemical or pharmaceutical product or for fluid clarification and recovery. A number of competing technologies and options can be employed to accomplish these steps, including nutsche filters, centrifuges, belt filters and others. This article concentrates on the testing of pressure or vacuum operations.

In solid-liquid separation systems, a wide variety of parameters influence performance. Evaluation and testing procedures can help plants determine the effectiveness of a particular system. Parameters that can be evaluated include particle size and shape, particle type, density, concentration, viscosity, cake height, pressure or vacuum, filter media, batch or continuous operation, required production throughput and more.

Theoretical calculations of filtration performance (Darcy's Equation and other modeling techniques) are far from easy, but can be useful. Creative problem-solving, however, continues to be a primary task of process engineers. The selected internal or external filtration testing personnel must have the ability to combine theory and practice.

BHS Pocket-leaf filter testing

Bench-top testing first must be used to narrow the gap between theory and practice and to begin the equipment selection process. A useful bench-top filter system is a BHS pressurized pocket-leaf filter (PLF), which resembles a Buchner funnel. The figure shown on the next page illustrates a typical PLF unit.

The PLF shown has a filter area of 0.002 m^2 and consists of a pressure vessel (90 psig to full vacuum), a top cover with a pressure gauge and gas (or air) connection and a bottom base for the filter media and filtrate outlet. The pressure vessel and base are jacketed and can be heated or cooled with a heat-transfer medium. The filter media can be a synthetic, single-layer metal or multi-layer sintered metal. The materials of construction are 316 Ti stainless steel, Hastelloy or polypropylene, and the fill volumes range from 250 milliliters (ml) to 2,000 ml.

A number of items are required for accurate PLF testing, including:

- Material Safety Data Sheets (MSDSs) for all materials.
- 4,000 ml to 8,000 ml of representative-quality feed material for each material to be tested.
- 2,000 ml of wash material for each wash.
- A 1,000 ml to 4,000 ml closed container with mixer to use for the feed material before each run.
- Several 250 ml to 500 ml containers for the feed material, the filtrate, the fresh wash material and the wash filtrates.
- Small containers for the filter cake.
- A gram scale.
- A vacuum oven or other technique 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 by a Karl-Fischer analysis or other 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. The flowmeter allows the air or gas flow rate to be measured during the drying step.
- A heat-transfer medium (hot oil, glycol, steam or cooling liquid).
- A vacuum source, for vacuum filtration.
- A specific test apparatus to measure data such as pH, conductivity, particle size after completion of the testing cycle, etc.

Representative Sample. The sample must be representative of what is to be found in the actual process, including particle size distribution, particle shape, viscosity, temperature, etc.

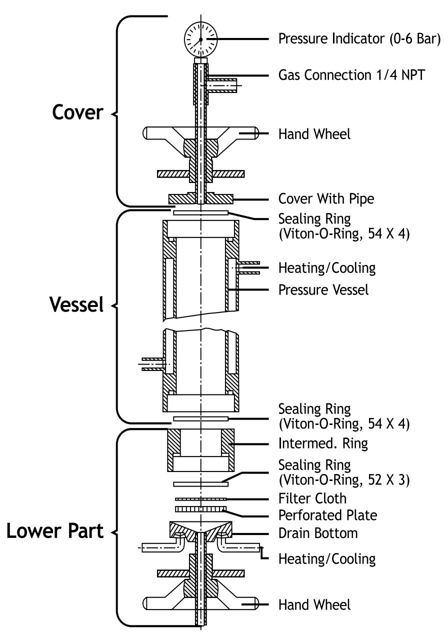
Testing location and personnel. Several options are available for the testing location. The first option is the plant's lab or pilot plant. This approach offers the best chance of a representative sample and provides easy access for all process engineers involved in the project. However, testing often will conflict with the plant's production requirements. Furthermore, time conflicts could exist, so it is important to determine who would conduct the testing at the plant site – the plant's or the BHS process engineers. If it is the BHS engineer, then safety training, laboratory access and other concerns must be addressed.

A second alternative is to conduct the tests at the BHS laboratory using process materials produced at the plant. This approach allows focused testing with little or no interruptions. In this case, it is important for the process engineer(s) to evaluate the vendor's laboratory, as well as the vendor's process personnel who will be conducting the tests. If possible, the plant's process engineer(s) should be invited to witness and help perform the tests; they will be familiar with the "quirks" of the process and product.

A third alternative combines the first two approaches at the BHS process and filtration laboratory. In this case, either the BHS or the plant process engineer(s) would supply the necessary process chemicals to conduct the reactions and/or precipitations. The resulting slurry then would be fed immediately to the PLF to begin the testing.

This approach offers benefits in that the sample is representative, the testing is focused, reaction/precipitation parameters can be modified to improve the filtration results, and a holistic approach to testing is implemented.

Required data and data collection. The testing objectives could be to expand plant production, decrease cycle times, maximize wash efficiencies or achieve another goal. Table 1 shows a typical data collection form that can be used for bench-top testing with the PLF unit. Table 2 illustrates the data about the process that are required, slurry, washing media and, most importantly, the testing objectives.



BHS Pocket-Leaf Filter

Table 1: Data Collection Form for PLF Tests

Customer: ____________ Test Number: ____________
Date: ________________ Test Unit: ________________

		Run #	Run #	Run #
	Filter Media"			
	Suspension:			
Filling	Volume of Slurry			
	Density of Slurry			
	% Solids in Food			
Filtration	Pressure			
	Temperature			
	Volume of Filtrate			
	Time for Filtration			
	% Solids in Filtrate			
	Wash Material			
Wash 1	Pressure			
	Temperature			
	Volume of Filtrate			
	Time for Filtration			
	% Solids in Filtrate			
	Wash Material			
Wash 2	Pressure			
	Temperature			
	Volume of Filtrate			
	Time for Filtration			
	% Solids in Filtrate			
	Wash Material			
Wash 3	Pressure			
	Temperature			
	Volume of Filtrate			
	Time for Filtration			
	% Solids in Filtrate			
Drying	Pressure			
	Temperature			
	Flow Rate			
	Time for Drying			
Cake	Weight			
	Thickness			
	% Residual Moisture			
	Discharge OK?			
	Cake rests on filter cloth?			

Testing procedures

Pressure or vacuum filtration. The first optimization is the filtration rate. A pre-measured amount of slurry is added from the top. Pressure or vacuum filtration begins, and the amount of filtrate vs. time is recorded.

Parameters that are varied sequentially in this step include cake depth, filtration pressure or vacuum and filter media. For thin-cake filtration technologies, cake depths can vary between 5 millimeters (mm) and 25 mm. Maximum cake thickness for the PLF unit is 150 mm.

Displacement washing. Displacement washing is performed after the filtration step is completed. A measured amount of wash liquid is added carefully in a predetermined wash ratio so the cake is not disturbed. Once again, pressure and time are measured. One or more wash tests can be conducted with the same or different wash liquids.

Cake pressing. The BHS thin-cake technologies can perform cake pressing or squeezing. The PLF can

Table 2: Application Data Information

Material: (Name of the suspension)	
	______________________________
I. Filter operation	1. The product is the: ____liquid ____solid ____both 2. What is the current method of filtration? ______________ 3. What needs to be improved? ______________
II. Production Rates	___Continuous 1. Suspension ______________m3/hr 2. Dry solids ______________kg/hr 3. Washing agent ______________m3/hr 4. Daily Production time__________hr/day OR ___Batch 1. Suspension/batch ______________m3 2. Dry solids/batch ______________kg 3. Washing agent/batch ______________m3 4. No. batches/day ______________ 5. Allowable batch time______________hr
III. Suspension	1. Density ______________g/l 2. Solids content ______________g/l 3. Average particle size______________micron 4. Temperature ______________deg. C 5. Viscosity______________m2/sec 6. pH______________ Composition a) liquid: ______________ b) solids: ______________ Type of solids: ___crystalline___amorphus___fibrous___colloidal
IV. Filter Cake	1. Desired residual moisture in the filter cake ______________% 2. Desired degree of washing ______________ 3. Permissible temperature for moisture determination _____deg. C 4. What happens to the filter cake when it is discharged? ________
V. Filtrate	1. Allowable solids content______________g/l 2. What happens to the filtrate when it is discharged? ______
VI. Washing	1. What wash agent will be used?_____________at___deg. C 2. Allowable quantity______m3/hr or______l/kg of dry solids 3. Required concentration of the filtrate produced________resp. 4. Will counter-current washing be required? ___no ____yes, with_____stages 5. What happens to the wash filtrate when it is discharged? ______________
VII. Recommended Materials of Construction	1. Metals: ______________ 2. Synthetic materials, elastomers: ______________ 3. Seals: ______________ 4. Filter cloth: ______________
VII. Comments	______________

simulate this pressing procedure with a "pressing plug." The pressing plug is actuated by nitrogen pressure and squeezes the cake onto filter media. This pressing can be conducted before, during or after the filtration, washing and drying steps.

Drying. Product drying in the PLF is tested by blowing ambient-temperature or hot gas through the cake or via vacuum. In addition, both the vessel jacket and base jacket are heated to simulate a production unit. The pressure is kept constant, and gas throughput is measured vs. time. After a preselected drying time, the cake is removed, and the cake depth and weight are determined. The cake then is analyzed for moisture content. Several iterations are required.

Results and analysis. Once testing is completed, the BHS process engineers analyze the data to recommend one or more filtration technologies. The test report includes an executive summary, test objectives, test methods and facilities, test data (in table form), test results (in written and graphical form, including filtration and drying curves), recommendations of production equipment and scale-up and any other recommendations and "path-forward" action steps.

Based on the PLF tests and recommendations, BHS pilot-scale tests can be conducted. These test should most often be conducted 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 also important for the plant to ensure that the BHS engineer who conducted the PLF tests conducts the pilot tests. The testing procedure and testing "tricks" employed on the bench-top, therefore, also will be employed in the pilot testing.

Conclusions

Currently, the most efficient approach to selecting and/or optimizing a pressure or vacuum filtration system is to use the BHS Pocket-Leaf Filter. With assistance and process support from BHS and accurate data from the testing – combined with filtration theory and experience – proper selection, scale-up, optimization and process guarantees can be realized.

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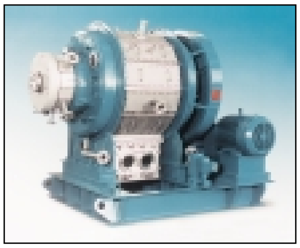
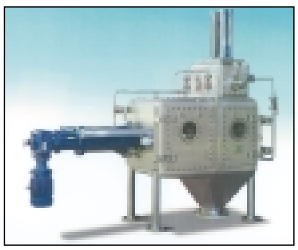
Specializing in Thin-Cake Filtration, Cake Washing & Drying Technologies

Test in the BHS Laboratory or at Your Plant for the Optimum Process Technology



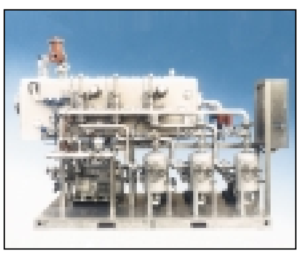
BHS Rotary Pressure Filter

- Continuous thin-cake (0.25-6 inches) production
- Filtration is conducted via pressure of up to 90 psig
- Positive displacement washing or counter-current washing
- Multiple washing steps as well as solvent exchanges, steaming and extraction
- Cake drying by blowing hot or ambient-temperature gas
- Atmospheric discharge from pressure operations for direct discharge to downstream equipment



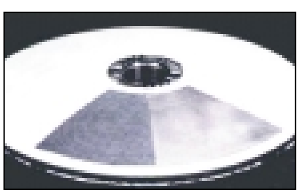
BHS Autopress

- The Autopress is installed in potent compound and active pharmaceutical ingredient (API's) facilities as well as for specialty chemical applications
- Thin-cake, typically 0.25-1.0 inches, production
- Filter plates are contained in a pressurized filter housing for complete containment
- Batch pressure filtration and forward or reverse flow washing
- Vacuum or hot-gas drying or pre-drying without agitation or tumbling
- Mechanical compression to 600 psig to eliminate cake cracking
- Fully automatic, heel-free and contained product discharge
- CIP systems with documented performance based upon Riboflavin tests



BHS Belt Filter

- Continuous filtration, washing and drying
- Thin-cakes between 0.25-4 inches
- Multiple washing zones for forward or counter-current washing to 99.99% purity
- Drying to 0.1% moisture with heated trays and mechanical compression
- Materials include stainless steel, Hastelloy and synthetic materials
- Gas-tight and pressurized housings



BHS Pressure Plate Filter

- Horizontally designed for stable cakes
- Possible pre-coating of the media with activated carbon or diatomaceous earth
- Vibrating plates, along with gas-assist pulsing, provide for automatic discharge
- Effective solids discharge without spinning-plates
- No rotating or mechanical seals

BHS Candle Filter & Inverting Filter

- Pressure filtration, clarification and heel filtration
- Specialized candles with perforated metallic or synthetic cores
- Filter media to less than 1 micron
- Cake washing produces a uniform cake for drying
- Cake discharge via gas blowback
- Possible pre-coating of the media with activated carbon or diatomaceous earth
- Cake discharge by inverting filter cloth for heel-free discharge and final moisture content to 1%.

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