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

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 m² 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.

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
perform cake pressing or squeezing. The PLF can

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

<table>
<thead>
<tr>
<th>Material: (Name of the suspension)</th>
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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
1. Continuous
   1. Suspension ______ m3/hr
   2. Dry solids ______ kg/hr
   3. Washing agent ______ m3/hr
   4. Daily Production time ______ hr/day

OR
1. 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
   a) liquid
   b) solids
   Type of solids: _____crystalline_____amorphous_____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 ______ kg/hr or ______ kg/1 of dry solids
3. Required concentration of the filtrate produced ______ %
4. Will counter-current washing be required? ______ 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

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.
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 (APIs) 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%.