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By Barry A. Perlmutter, Managing Director



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Solid-liquid separation and cake washing are integral steps in the production of bulk or final pharmaceutical products. Each step must be optimized, along with the downstream dryer.

Complicating this process is the number of competing technologies and options that can be employed to accomplish these steps. For example, most pharmaceutical processes will use either an agitated nutsche filter or a centrifuge for the separation and washing step.

This article discusses the use of the thin-cake Autopress pressure filter as an alternative to agitated nutsche filters and centrifuges. The results are applicable to specialty chemical and other applications with similar process crystals and requirements.

Autopress pressure filter

Autopress filter operation begins with slurry filling to form thin filter cakes—typically of a 5-millimeter (mm) to 25-mm thickness. Pressure filtration continues operating up to 150 pounds per square inch gauge (psig). The cake then can be mechanically compressed up to 600 psig to eliminate cracking and ensure maximum washing efficiency in the forward

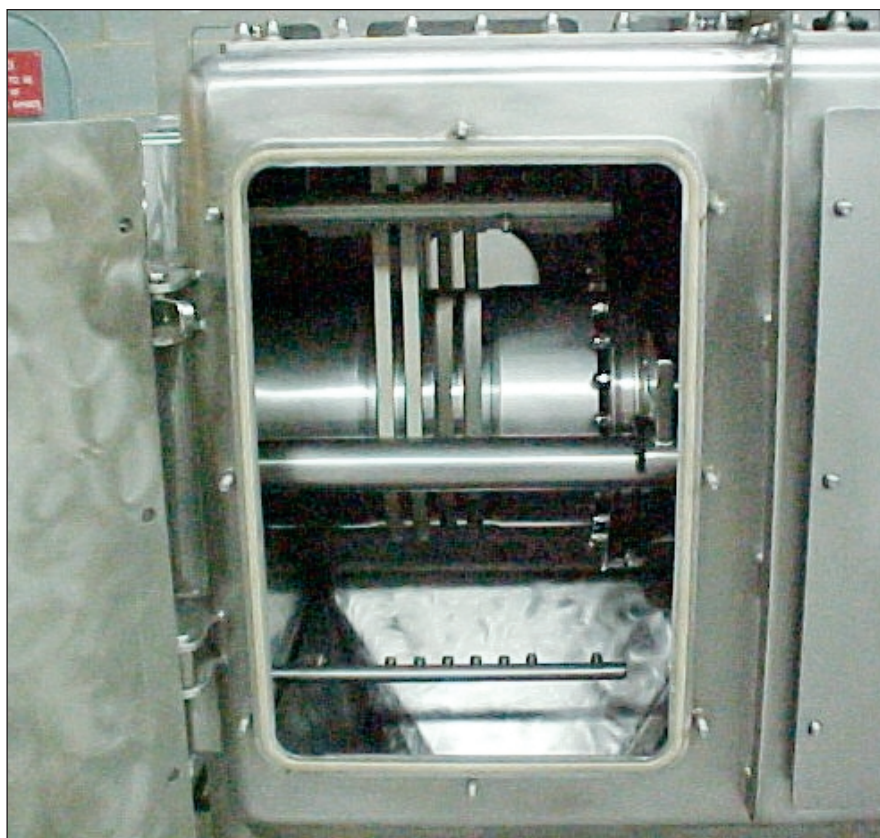


Figure 1. Autopress internals showing filter plates and knives for automatic and contained product discharge.

or reverse direction. Finally, the cake can be pre-dried or fully dried by either introducing vacuum or blowing gas through the cake in either direction.

Final moisture contents of less than 0.1 percent are possible. This gentle drying without agitation or tumbling is important for fragile

crystals and thixotropic cakes. Elastomeric knives sequentially and automatically discharge the circular cakes before the filter begins a new cycle, Figure 1.

Taking the technology to task

Recently, a new pharmaceutical

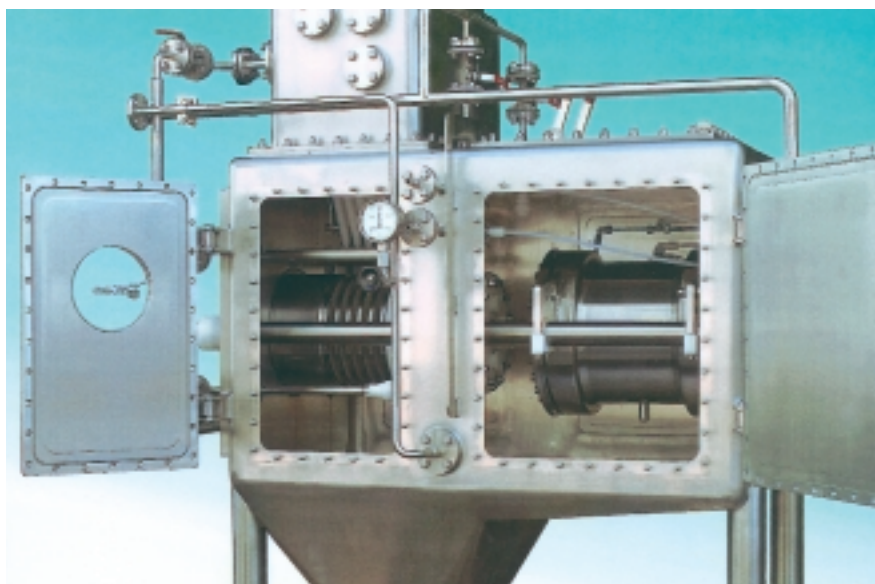


Figure 2. Autopress internals showing filter plates and pressurized filter plate housing.

biomass product was being developed at a specialty chemical facility. The process steps included mixing with methanol, filtration and washing. The final dryness specification was less than 20 percent before the product was directly discharged into the downstream dryer.

Initially, the plant engineers examined nutsche filtration and centrifugation. The initial testwork on agitated nutsche filters and centrifuges produced very long cycle times and low washing efficiencies. The filtration and washing flux rates were much slower than that required to meet the batch times.

The product crystals are “straight” in shape and, as a result, tended to pack together, resulting in a low-permeability cake. For a nutsche filter to be effective, cake depth typically is greater than 150 mm. In this case, the filtration rate was very slow at a depth greater than 25 mm.

In the centrifuge, these needle-like crystals—as a result of the high g-forces—also resulted in plugged filter cloth and low permeability

for filtration and washing. Based on these results, the plant engineers decided to investigate a filtration technology that would allow pressure filtration with a cake depth of less than 25 mm.

The Autopress technology consists of specially designed vertical circular filter plates with synthetic or sintered metal filter media. The filter plates are sealed to each

other using specially designed U.S. Food and Drug Administration (FDA)-approved elastomers to eliminate solid, gas or liquid bypass. The elastomeric spacers, unlike nutsche filters or centrifuges, also ensure absolute reproducible cake thickness.

The filter plates are contained in a pressurized filter housing, where a gas-inflated membrane seals the annular space between the housing and the filter plates, shown in Figure 2. All operations are contained from full vacuum to 150 psig. The entire filter housing is enclosed in a pressure-tight outer housing for complete product containment, Figure 3.

Autopress filter testwork

To evaluate an Autopress filter, the initial testwork generally is conducted using a pressurized pocket-leaf filter with 20 square centimeters (cm²) of filter area, Figure 4. These tests can be conducted either in the customer’s lab or in the manufacturer’s laboratory. The pocket-leaf filter is used to gather the basic filtration, washing

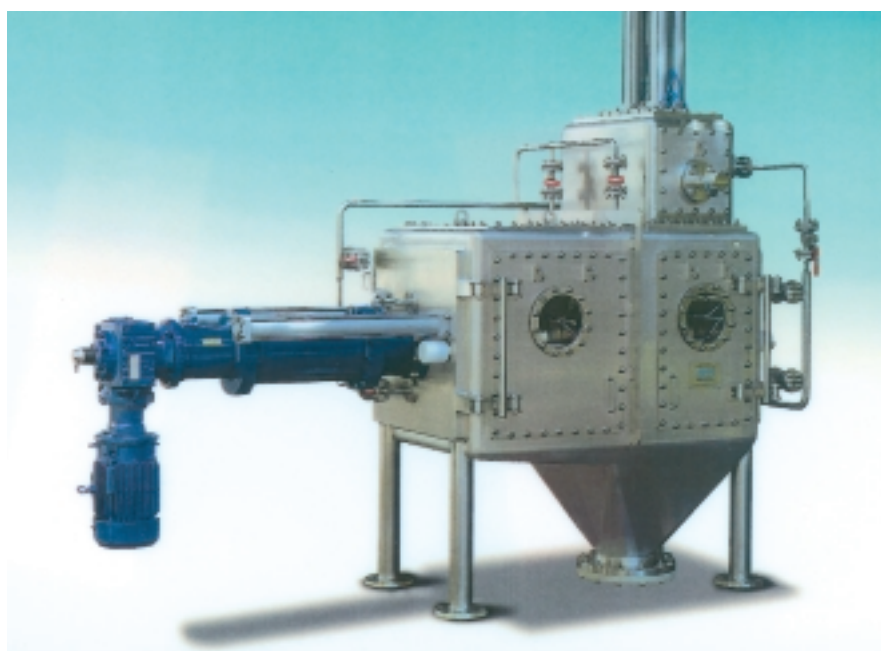


Figure 3. Autopress for complete product containment.



Figure 4. BHS Pocket-Leaf Filter for bench-top testing for filtration, washing and drying studies.

and drying data.

In the pharmaceutical biomass application, the product was mixed with methanol, filtered and washed. The final dryness had to be less than 20 percent before the product was directly discharged into the downstream dryer. The slurry was prepared with dry product and mixed with cold methanol (less than 20° C) for 30 minutes. Slurry density was 180 grams/liter. The filter media tested—a sintered-metal screen—had a removal rating of 2 microns (μm) to 5 μm .

Filtration. The first optimization concerned the cake depth vs. the filtration rate. Filtration was conducted via pressure. A premeasured amount of slurry was added from

the top, and the unit was pressurized. When the filtration began, the amount of filtrate vs. time was recorded at constant pressure. Other parameters that were varied sequentially include cake depth, filtration pressure and filter media. As stated earlier, cake depths can range between 5 mm and 25 mm.

Washing. Displacement washing tests also were performed in the pocket-leaf filter. For accurate testing, it was necessary to ensure the cake did not crack before washing. To accomplish this, a pretest was conducted to determine the amount of filtrate that is displaced before nitrogen breakthrough. Once this was determined, the washing tests began. Filtration

was conducted until the filtrate quantity was met. Then, a measured amount of methanol wash liquid was added carefully in a predetermined wash ratio so as not to disturb the cake. Once again, pressure and time were measured.

Cake pressing. The Autopress filter can perform cake squeezing, as previously described. To simulate the pressing procedure, a “pressing plug” was used with the pocket-leaf filter. The pressing plug was actuated by nitrogen pressure and squeezed the cake onto filter media (this can be accomplished either before or after washing or before drying).

Drying. Product drying in the pocket-leaf filter was tested by blowing ambient-temperature or hot gas through the cake. The pressure was kept constant and gas throughput was measured vs. time.

After a preselected drying time, the cake was removed from the pocket-leaf filter. The cake depth was determined, and the cake was weighed and analyzed for moisture content. After several iterations, the drying times were optimized along with gas pressure and flow rate to achieve the better-than-20-percent final solvent content in the cake.

Results

Table 1 contains the test data and shows the process times, pressures and results. The pocket tests illustrate that the Autopress pressure filter can yield the required product specifications.

Filtration, washing, pre-drying and discharge times result in a cycle time of 20 minutes. Residual moisture can be achieved via cake compression and gas blowing. The

Laboratory Data and Scaleup

Suspension: Biomass Product
Washing liquid: Cold Methanol (less than 20°C)
Requirements:
1. Complete batch is to be processed in six (6) hours
2. Residual methanol to be less than 20 percent
3. Product fines in filtrate should be "no detectable losses"

	Trial 1	Trial 2
Filtration		
Pressure	14.7 psi	14.7 psi
Temperature	28°C	20°C
Volume of mother filtrate	140 ml	350 ml
Time for filtration	40 sec.	40 sec.
Washing		
Pressure	14.7 psi	14.7 psi
Temperature	20°C	20°C
Volume of washing liquid	20 ml	50 ml
Time for washing	7 sec.	7 sec.
Drying		
Pressure	30 psi	45 psi
Temperature	Ambient	Ambient
Flow rate	Not measured	Not measured
Cake pressing	No pressing	90 psi
Time for drying	2 min.	10 min.
Cake discharge		
Ease of discharge	Easy	Easy
Cake		
Weight	25.6 g	52.7 g
Thickness	12 mm	25 mm
Residual moisture	26 percent	14.8 percent
Discharge OK?	Yes	Yes
Cake cracking?	No	No
Cake rests on filter cloth?	No	No

Table 1

filtrate is visually clear, and product fine losses are less than 0.1 percent. Cake discharge appears easy without any sticking.

The required batch size is 3.5 cubic meters (m³) of cake. The specific capacity from the test results shows 70 liters of cake/square meter (m²) of filter area. The cycle time scaleup was approximately 11 minutes. When filling, discharge and miscellaneous activities are added in, the result was a 20-minute cycle time, from Table 1.

The customer requirement was to complete a reactor batch in six hours, which leads to a Autopress filter with 3.0m² of filter area (70 liters [l] of cake/m² x 3m² x 3 cycles per hour [each cycle is 20 minutes] x 6 hours=3,780l or 3.780m³).

Pilot-scale testing

Based on the successful pocket-leaf filter results, the chemical plant's process engineers decided

Autopress Filter Process Operations

The Autopress process operations begin with filling and filtration. Filling is either via the bottom of the plates or top and bottom for fast setting products. Cake washing then can be in the forward or reverse direction.

Cake pressing can be used to prevent cracks from developing in the cake. Cake drying is, as cake washing, in the forward or reverse directions and with or without cake squeezing. The cake is discharged and then, depending on the product requirements, a CIP operation can occur or the filling cycle can begin again.

The six process modules can be run as a manual, semi-automatic or fully automatic sequence. The PLC provides the choices to the operator with feedback to the plant's distributed control system. Figure 5 shows a typical P & ID for an Autopress installation.

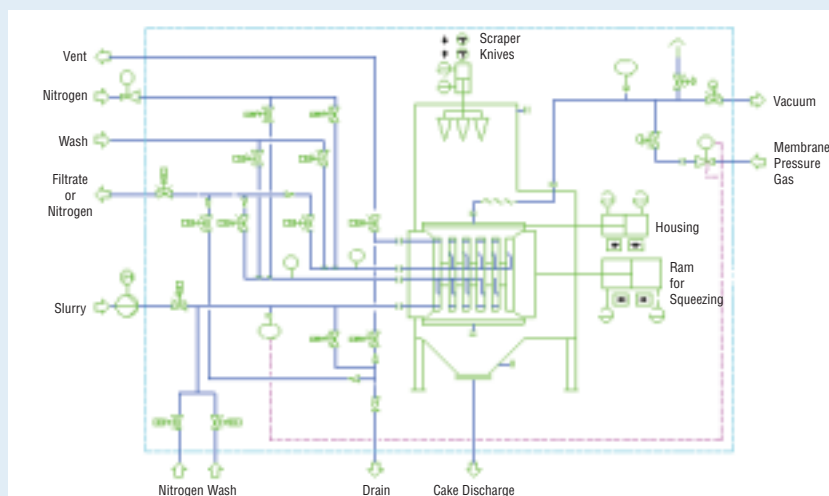


Figure 5. Typical P & ID for an Autopress Installation.

to continue the test program with a pilot-scale Autopress filter unit with 0.4m² of filter area. The pilot unit is controlled by a programmable logic controller (PLC) and fully automated, which allows for actual pilot-scale operation and is similar to a production-size unit.

Initially, six test cycles were run. After optimization, 31 cycles were achieved during the testing.

Pilot-scale results

Filtration:

- The average filtration rates compared favorably to the bench-scale test work.
- Feed concentration did not impact the filtration rates.

- Product fines and losses in the filtrate were minimal.

Note: The plant determined that a mass flow measurement was important to ensure the plates are completely full of product for effective filtration and washing.

Solids behavior. Heavy solids have a high settling velocity and can exhibit preferential buildup between the plates. The Autopress filter was designed for special filling techniques to overcome the high settling velocity. This is an advantage the Autopress has over centrifuges and nutsche filters.



Figure 6. BHS PLC Control System.

Washing:

- For maximum washing efficiency, the plates must be full.
- Cake compression is important to eliminate cracks that can form in the cake.
- The ability to compress or squeeze the cake at different times in the process sequence, as well as at different squeezing pressures, is an important process benefit.
- Two displacement-washing steps were incorporated into the process with squeezing between the steps; forward and reverse washing were employed.
- Cake analyses showed low and/or nondetectable impurities after the two washes.
- The washing pressure was higher than the filtration and feeding pressures. These process capabilities can be incorporated into the PLC program for automatic operation.

Drying:

- Drying and final moisture levels were impacted by nitrogen flow rate, which was varied

between 5 standard cubic feet per minute (scfm) and 20 scfm.

- Nitrogen gas temperature, which was varied between ambient and 60°C, had little impact on the final moisture.
- Nitrogen blowing time had the most impact and was varied between 5 min. and 40 min.
- Gas pressure was held constant—generally below 30 psig. This was important for the fragile crystals.
- Channeling through the cake could occur, but cake pressing eliminated this. This would be an advantage over nutsche filters.



Figure 7. BHS Process Piping, Valves, and Instrumentation Skid to complete the Autopress Installation.

Discharge:

- Material discharged easily by gravity and with the scraper knives.
- Product heel was minimal, and in some cases was eliminated.

Conclusion

Side-by-side tests were conducted with an agitated nutsche filter, centrifuge and the Autopress pressure filter. Based on the laboratory and pilot-plant tests, the Autopress pressure filter was the technology selected for the new process. The plant installed a total of two Autopress filters for the bulk intermediate and the final product.

All products wetted parts were polished stainless steel with rounded corners to eliminate product holdup. The seals consisted of all FDA-approved materials. The filter media selected was a 2- μ m sintered stainless steel that is welded to the filter plates. All process and mechanical operations were either pneumatic or electric, allowing a clean installation.

BHS provided the total process solution including a PLC system, according to GAMP guidelines, shown in Figure 6. The process piping, valves, and instrumentation skid, in Figure 7, completed the installation.

Finally, as part of the Factory Acceptance Test (FAT) and Site Acceptance Test (SAT), a Riboflavin clean-in-place (CIP) test was conducted to demonstrate the ability of the Autopress Filter to be cleaned for batch-to-batch integrity or product-to-product campaigns. Photos 8, 9, and 10 show the CIP test results. The units have been installed and validated and are meeting the production requirements of the plant.

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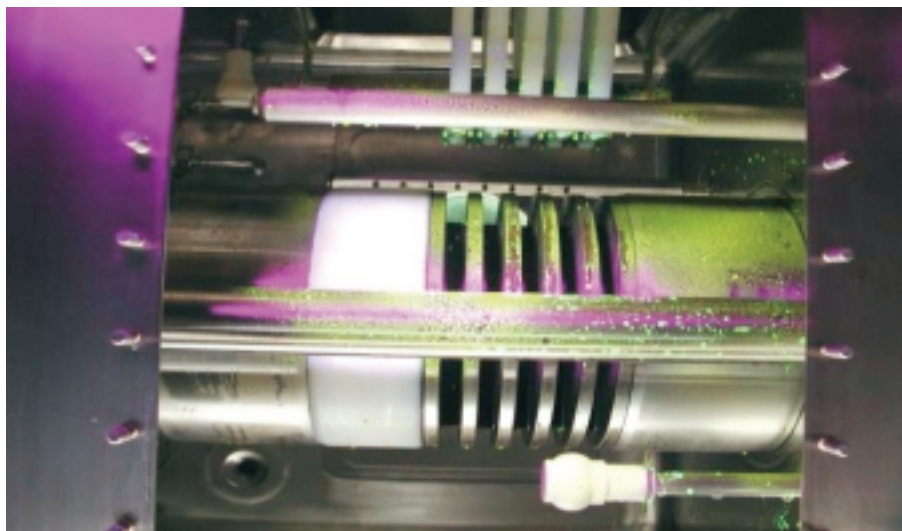


Figure 8. BHS Autopress beginning CIP Test with Riboflavin. View into the contaminated process area.

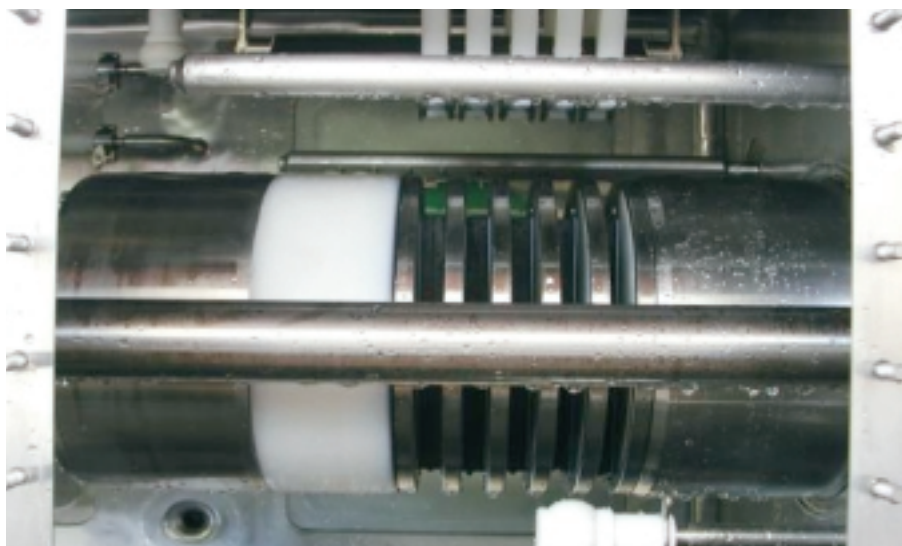


Figure 9. View into the process area after CIP cleaning.



Figure 10. View into the discharge knife area after CIP cleaning.

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

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



BHS FEST 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



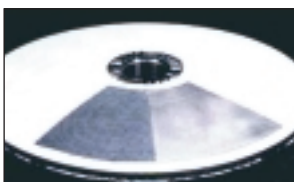
BHS-Autopress

- 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

- 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

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