PHARMACEUTICAL PRODUCT RECOVERY USING BHS CONCENTRATING CANDLE FILTERS DOWNSTREAM OF CENTRIFUGES[1]

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INTRODUCTION

In the present process, the pharmaceutical slurry goes to a centrifuge for separation and washing and the cake is discharged as the product. The centrate of the centrifuge is discharged as a waste material. However, the centrate contains soluble product which is presently lost. The purpose of this project is to recover this material.

The proposed system is to concentrate the soluble material followed by precipitation of the product. The slurry is then further concentrated using a BHS candle filter with the concentrate being introduced back to the process.

After extensive testing with the BHS PLF test filter (discussed below) and discussions with the customer, the BHS candle filter was installed. The unit is producing concentrated slurry of 10% from an initial concentration of 1%. The process was then adjusted to concentrate a 3% slurry to 10% this was easily accomplished by adjusting the filtration timer. The design of the BHS candle filter followed cGMP guidelines including the qualification documents.

PROCESS OBJECTIVES

The pharmaceutical (Pharma) Company had determined that the centrate of the centrifuge contained valuable product that was in solution. The material is concentrated using a membrane process, the soluble material is then precipitated by adjusting the slurry pH. This proved to be successful and the next step was to determine how to recover and concentrate the solids. The pharma company contacted BHS for process consulting to determine the optimum method of filtration-concentration.

[1] **Author wishes to thank Bruce Glines, BHS Senior Process Engineer, for his test work, project, start-up and commissioning contributions to this article.**

BHS SONTHOFEN

BHS PROCESS CONSULTING FOR FILTRATION – CONCENTRATION

The Pharma Company and BHS brainstormed various solutions such as hydrocyclones, settling tanks, cross-flow membranes and determined that the filtration-concentration process could be accomplished by either candle filters or pressure plate filters, which are both batch-operated, pressure-filtration systems. The cake structure and other parameters would determine which system is appropriate for the application.

CANDLE FILTRATION SYSTEMS

A candle filter is a pressure vessel filled with tubular filters called candles (Figure 1). candleThe candle is comprised of a filtrate pipe, 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 micron (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, alloys, or in some cases lined with a coating. Within the vessel are horizontal manifolds called 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 filtration time is reached. The cake is then washed to remove impurities and residual mother liquor finally, the cake is dried.

For cake concentrating, low-pressure gas enters in the reverse direction through the registers and into 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 where the concentrated slurry is collected.

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.

PRESSURE PLATE FILTRATION SYSTEMS

Similar to the candle filter, pressure plate filters (Figure 4) are comprised of filter plates, contained within a pressure vessel. However, instead of vertical filter candles, the vessel contains horizontal filter plates.

These plates are slightly sloped, conical-shaped metal plates that support a coarse-mesh backing screen covered with filter cloth (Figure 5). An opening in the center of the plate allows the filtrate to travel between plates and out of the vessel.

The operation is similar to a candle filter. The slurry enters the bottom of the vessel and is pumped upward. The solids build up between the plates, while the liquid flows through the core of the filter plates and exits from the top of the vessel. The cake is then washed and dried. Two unbalance motors vibrate the filter plates to dislodge the cake from the filter cloth so it can be discharged (Figure 6). The concentrated slurry collects in the cone.

Pressure plate filters are used for filtration of cakes greater than 20 mm thick. They are selected for cakes that are stable horizontally because of the orientation of the plates.

BHS PROCESS CONSULTING TECHNOLOGY SELECTION

The cake structure and process parameters determine which system is optimum for the application. Cakes that are stable vertically are compatible with candle filters, whereas cakes that are stable horizontally are best handled by a pressure plate filter. For example, very dense cakes or very fluffy cakes would be better processed on a pressure plate filter.

The candle filter is limited to cake structures with thicknesses between 5 mm and 20 mm, while a pressure-plate filter can handle thicker cakes greater than 20 mm. Both units can conduct filtration up to 150 psig.

Candle and pressure plate filtration can remove 1–3-µm particles. In some applications, candle filters can remove even finer particles, as small as 0.5 µm. Both systems use synthetic filter media, but pressure plate filters can also use metal media for high-temperature applications (greater than 200°C) or if steaming is required.

If the process requires washing of the solids, a pressure plate filter is preferred, because the horizontal orientation eliminates the possibility of the cake falling off the plate during the draining operation. When washing is not critical, a candle filter may be the best choice for clarification and removal.

Drying of the cake can be achieved on both filters, this can be accomplished through the continuous flow of drying gas or through pulse drying. The candle filter can also be used as described above as a concentrator, with a slurry.

Clean-in-place (CIP) operations can be conducted similarly in both systems. The vessel is filled with cleaning fluid that is circulated while gas is blown in the reverse direction, which creates a turbulent mixture that has a quasi-ultrasonic cleaning effect. Cleaning of pressure plate filters is enhanced by the ability to vibrate the plates.

DETERMINING FILTRATION – CONCENTRATION SYSTEM DESIGN

Laboratory testing at a constant slurry flow or constant pressure is used to determine the size and design of the system for processes with extremely low concentrations of solids. The test evaluates the filter media, operating pressure, and cake thickness to determine the optimum clarification system design and size.

A lab-scale Pocket Leaf Filter (PLF) shown in Figure 7 is used as the test filter. Various filter media can be installed, depending on the desired filtrate clarity, filtration flux rate (time), cake thickness, and cake discharge rate, as well as compatibility with the process. Constant volume tests use a peristaltic pump to supply a constant flow of slurry to the PLF and a pressure gauge measures the change in pressure across the filter as the cake builds.

During the testing, cake thickness was very thin at 7 mm which would lend itself to a candle filter decision. Further, as there was no cake washing or drying, but only concentrating, this also pushed the decision towards candle filters. The filtration results are shown in Figure 8. The particle size distribution has a mean of 4 microns (um) and the chosen FDA-approved filter media produced a clean filtrate which was then used back in the process. The cake discharged from the filter media easily and was able to be concentrated in the cone.

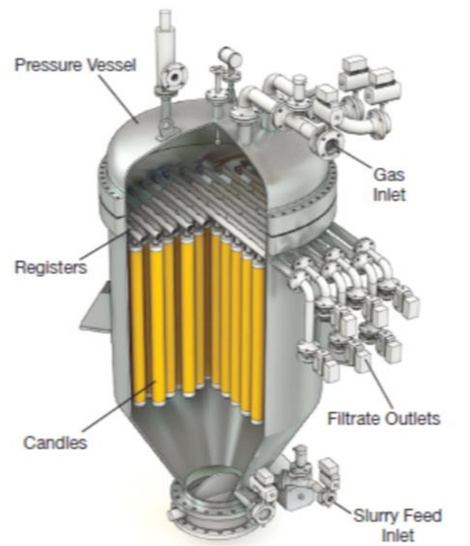
BHS PHARMA CANDLE FILTER INSTALLATION

BHS installed the concentrating candle filters, shown in Figure 9, as a complete turnkey skid package including piping, wiring and PLC controls. The candle filter is manufactured to cGMP design with PLC controls meeting the ISA-S88 requirements. BHS conducted the riboflavin testing demonstrating CIP compliance with the Pharma Company's quality control group and assisted with the IQ/OQ documentation.

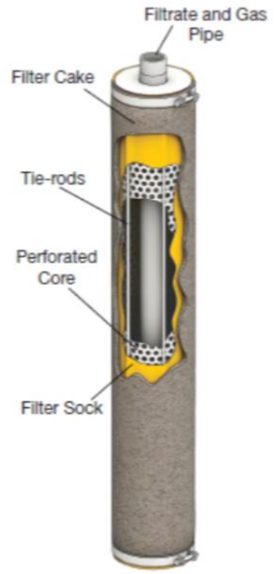
The final pharma process includes slurry filling, filtration timer to process a required volume of material. The vent valve is opened to release pressure, the discharge valve is opened and the contents are discharges to the process feed tank. A back wash is provided to clean the media and ensure a complete discharge of solids. The solids concentration is monitored to ensure a consistent feed back to the process. If adjustment is needed the filtration time can be changed as required. The process rate has met the customer's expectations.

TAKEAWAYS

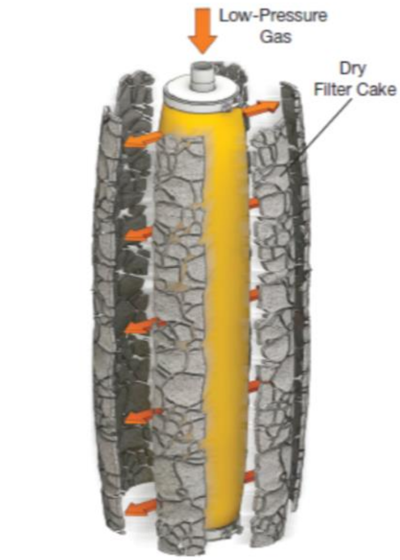
There are many choices of technologies for fines filtration-concentration or as a dry cake discharge. The newer approaches of candle filters and pressure plate filters provide for higher quality filtration, efficient concentrating and improved yields with fully automated and contained operations. These systems are also installed for catalyst recovery or concentration as well as in a concentrating mode in combination applications. Creative thinking with BHS process consulting provide for optimum production results.



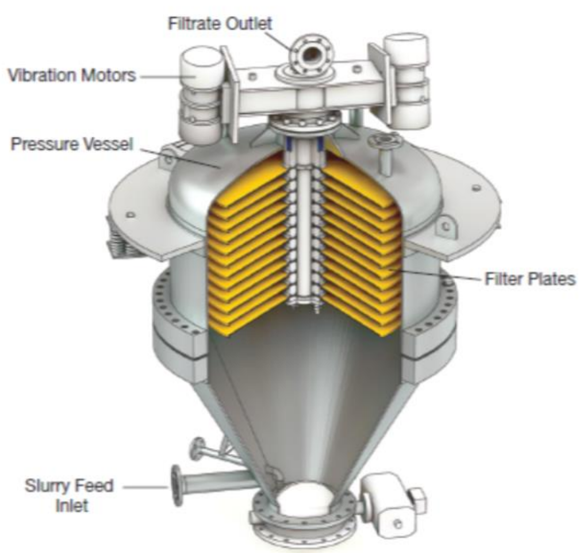
▲ **Figure 1.** In a candle filter, the slurry enters through the ottom of a pressure vessel and flows across the filter media. he filter candles are attached to registers that collect the ltrate. Gas is fed into the top of the pressure vessel for cake rying and discharge.



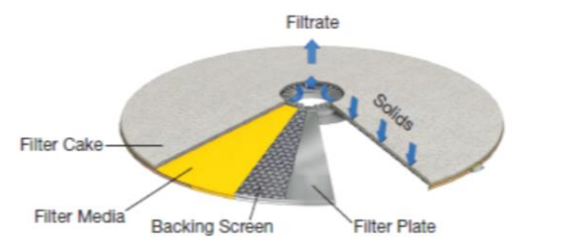
▲ **Figure 2.** During operation, filtrate exits from the top of the candle, while the solids collect on the synthetic filter sock.



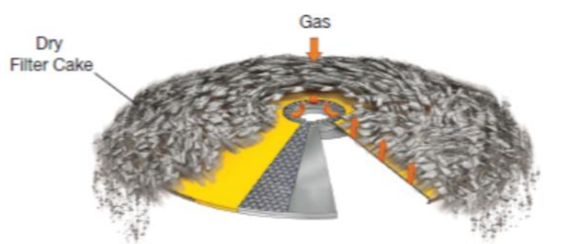
▲ **Figure 3.** During discharge, gas is fed into the top of the candle, which expands the flexible filter sock. This causes the dry cake to crack and break away from the filter. The solids are collected at the bottom of the pressure vessel.



▲ **Figure 4.** In a pressure plate filter, the slurry is pumped at the bottom of a pressure vessel. The solids collect on the filter medium, while the filtrate flows through the core of the plates and exits through the top of the vessel. Two unbalanced motors attached to the top of the unit effect cake discharge.



▲ **Figure 5.** Each filter plate is covered by a backing screen and filter medium, which may be metal or a synthetic. The solids accumulate on the surface of the plate, while the filtrate flows through the core.



▲ **Figure 6.** After drying, the cake is discharged from the plates by vibration created by two unbalanced motors affixed to the top of the vessel. Gas is fed from the top of the unit to help push the dry cake off the plates and to the bottom of the pressure vessel.

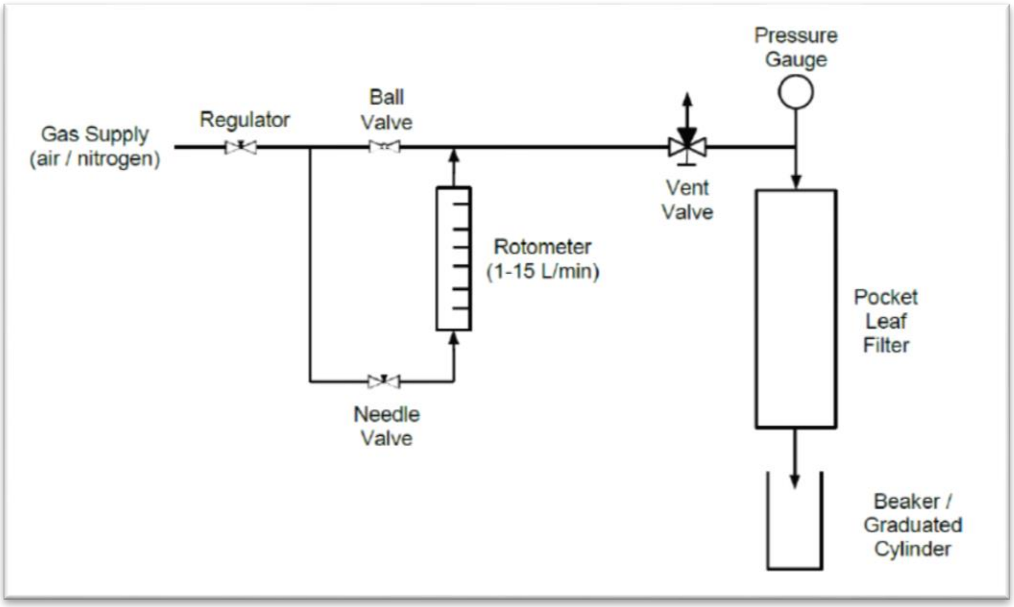


Figure 7: BHS Pocket Leaf Filter for Pressure Testing

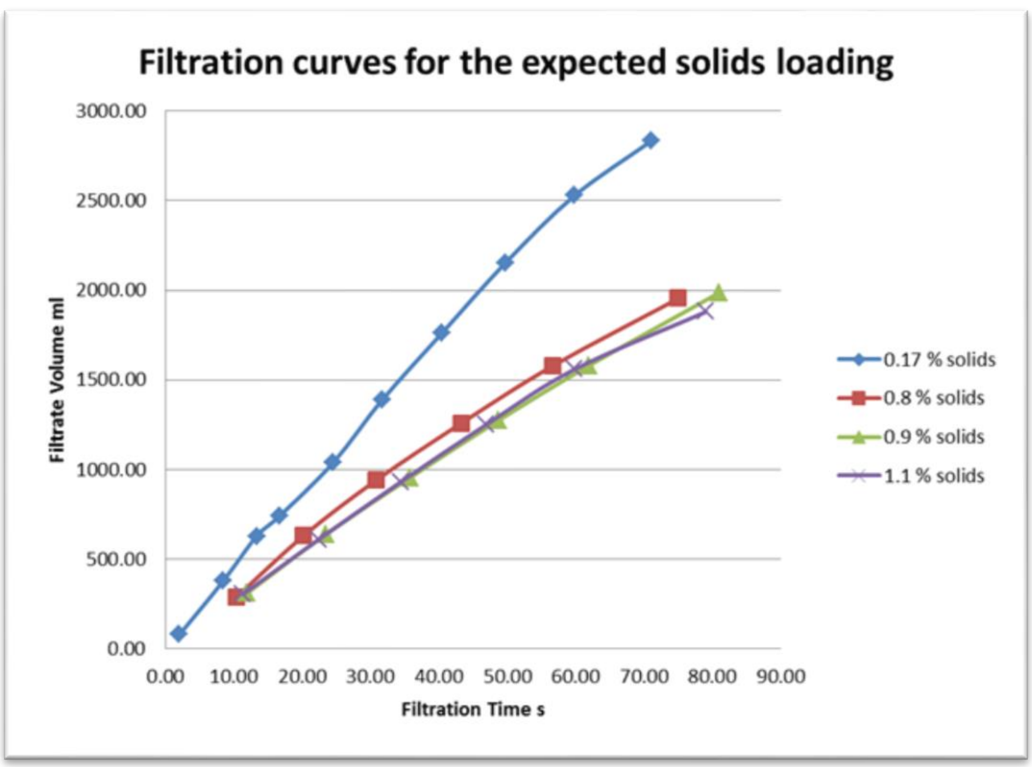


Figure 8: BHS Process Filtration Curves

Figure 9: BHS Pharma Candle Filter for Concentrated Slurry Discharge