New Approaches for Removing Catalyst Fines From Chemical and Pharmaceutical Slurries[1]

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Introduction

Many chemical, petrochemical and pharmaceutical manufacturing processes involve reactions of solid- and liquid-phase reactants to produce a slurry. The slurry typically needs to be separated into its component parts — the mother liquid and the solid.

The valuable material may be the liquid, the solid, or both phases. The location of the valuable component to be recovered determines the type of separation method and equipment used. For example, if the liquid is the desired product, the solids are removed to produce a clean liquid; if the solid is the desired product, removal of small particles increases the product yield.

Generally, the first step after the reaction is a bulk separation process that removes large, coarse solids (larger than 5 μm). This is relatively straightforward, but as processes have become more sophisticated and quality requirements have tightened, it has become necessary to remove residual particle fines from slurries. These particles are small — typically 1–5 μm in diameter, or even smaller — and at low concentrations, on the order of parts per million (ppm). This article describes a new approach to clarification.

Coarse Particle Filtration

The choice of a filtration technology (clarification or coarse) is influenced by the process and the nature of the product. For example, coarse filtration may not be necessary if the product is a liquid and the solid byproducts are smaller than 5 μm; instead, clarification technologies can be employed exclusively. Alternatively, if the product is a solid and the reactor produces particles with a wide size distribution, coarse filtration maybe necessary.

Coarse filtration systems may operate as either batch or continuous processes, under the influence of gravity, pressure, vacuum, or centrifugal force. The method of operation for the filtration system depends on the type of reactor used, as well as the type of equipment downstream, such as a batch or continuous dryer.

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Existing Clarification Systems

A clarification system is employed after coarse-particle or as a stand-alone system to remove fine particles at low concentrations.

Currently, most slurries are clarified with the use of manual plate filters, filter presses, bag filters, cartridge filters and other conventional filter equipment. All of these units require manual operations for cake discharge and cleaning between batches or campaigns as well as suffer from high labor and maintenance costs, high disposal costs and the exposure of the operators and the environment to toxic and hazardous solvents and solids as well as used and contaminated filter cloth, bag filters and filter cartridges.

New Approaches to Clarification Systems

This is accomplished by candle filters and pressure plate filters, which are both batch-operated, pressure-filtration systems. The cake structure and the nature of the process determine which type of clarification system is appropriate for an application.

Candle Filtration Systems

A candle filter is a pressure vessel filled with tubular filters called candles (Figure 1). The 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 discharge, 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. The cake can also be discharged as a concentrated slurry.

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 that of 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).

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.

Clarification Technology Selection

The structure of the cake determines which system is better suited for the application. Cakes that are stable when oriented vertically are compatible with candle filters, whereas cakes that are stable when horizontal 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 operation. When washing is not critical, a candle filter may be the best choice for clarification and removal, since the cake is normally disposed of as waste.

For applications that require cake drying, pressure plate filters are appropriate because they create cakes that are as dry as possible without the application of heat. Candle filters produce less dry cakes, but this value depends on the specific cake material.
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 Clarification System Design
Laboratory testing at a constant slurry flow or constant pressure is used to determine the size and design of a clarification 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.

Wash analysis consists of running a liquid through a peristaltic pump, or adding wash liquid to the leaf filter and applying air pressure until the target quality (conductivity, acid level, etc.) is reached.

The cake is then dried with compressed air or nitrogen. The amount of compressed gas is regulated to prevent the use of excess air, which could lead to overly optimistic moisture result and would require air uneconomical volumes of air when scaled up. Alternatively, if concentrated slurry is required, then a known about of liquid is added to the leaf filter to simulate the slurry discharge.

Once the filtration tests are complete, the filtration the least-squares analysis is used to define the line through the data, with constants *m* and *b* determined by slope and y intercept of the line.

$t = m\,(V/A)^2 + b$ Equation

t is the filtration time (min), m is a derived constant (m^4-min/m^6),
V is the feed volume (m^3), A is the area of the leaf filter (m^2), and
b is a derived constant (min).

After rearranging, the area of the filter, *A*, can be determined from the feed volume, *V*, and the filtration time, *t*.

The tests provide the times for various process steps (washing, drying). Estimates are used for filling and draining times and the sum of the times gives the overall cycle time. The required production over this period is then used to calculate the required area.

Raney Nickel Catalyst Application with Candle Filters for Slurry Discharge
In this application, the current process after the reactor is gravity separation, hydrocyclones and then followed with cartridges and bag filters. The specification for the process liquid (diamine and water) is less than 3 ppm catalyst. This recovery process was inefficient and exposes the operators to the diamine and catalysts creating a safety hazard. The average particle size is 2 um and amorphous crystals.

Lab testing and pilot testing, shown in Figure 8, was conducted to determine a processing scheme that eliminates solvent exposure, reduces the maintenance and operation requirements of the current scheme and recovers the catalyst to less than 3 ppm. Current production throughput is a maximum of 185 gpm. Figure 9 shows the final design of a BHS slurry-discharge candle filter with 19 m^2 of filtration area.

Pharmaceutical Hydrogenation Application with Candle Filters for Dry Cake Discharge
In this application, the current process after the reactor is metal bag filters for slurry discharge into manual nutsche "clamshell" filters for vacuum filtration and drying. The process was very time-consuming and required handling of liquids and solids including final "manual dig out" of the nutsche filters. The process solvent was tetrahydrofuran (THF) and ethanol.

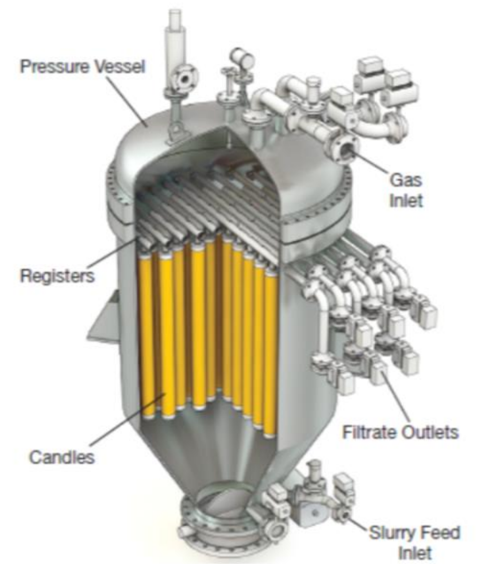
Lab testing was conducted to develop the one-step process for filtration and drying. The BHS candle filters, drawing shown in Figure 10, with pharma designed candles and cGMP compliance allowed for a revamp of the operation with two filters, one-on/one-off, each with 2 m^2 of filtration area.

Palladium Catalyst Filtration, Washing and Drying with a Pressure Plate Filter

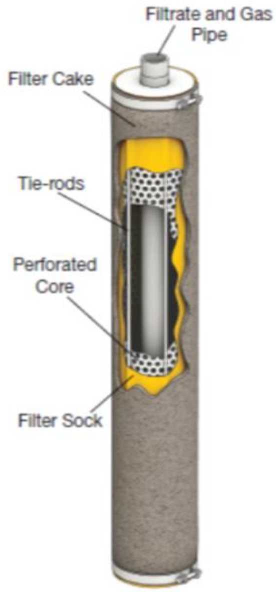
In this application, manual filter presses were used to recover and reuse the palladium catalyst. The filter presses exposed the operators to the process and had inefficient washing and drying. The process had a very short cycle of 4 hours per batch for the complete operation of 6 m^3 of slurry with 60 kg solids. The BHS pressure plate filter with 6 m^2 of filtration area, shown in Figure 11, completes the filtration, two-step cake washing, nitrogen blow drying and cake discharge in less than 4 hours with full containment.

Takeaways

There are many choices of technologies for fines recovery. The newer approaches of candle filters and pressure plate filters provide for higher quality filtration, improved yields with fully automated and contained operations. So, although they have an initial higher capital cost compared with manual systems, they result in a more reliable, efficient, and optimized process.

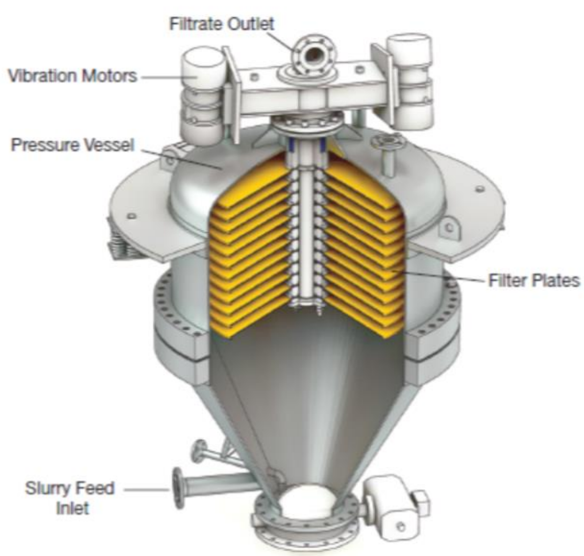


▲ **Figure 1.** In a candle filter, the slurry enters through the ottom of a pressure vessel and flows across the filter media. he filter candles are attached to registers that collect the ltrate. Gas is fed into the top of the pressure vessel for cake rying and discharge.

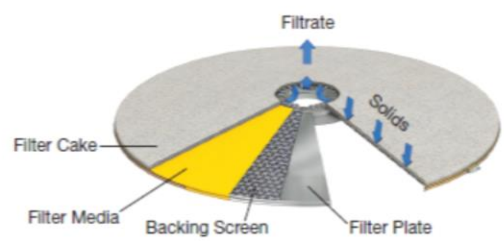


▲ **Figure 2.** During operation, filtrate exits from the top of the candle, while the solids collect on the synthetic filter sock.

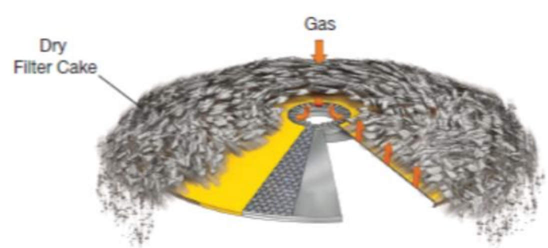
▲ **Figure 3.** During discharge, gas is fed into the top of the candle, which expands the flexible filter sock. This causes the dry cake to crack and break away from the filter. The solids are collected at the bottom of the pressure vessel.



▲ **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.

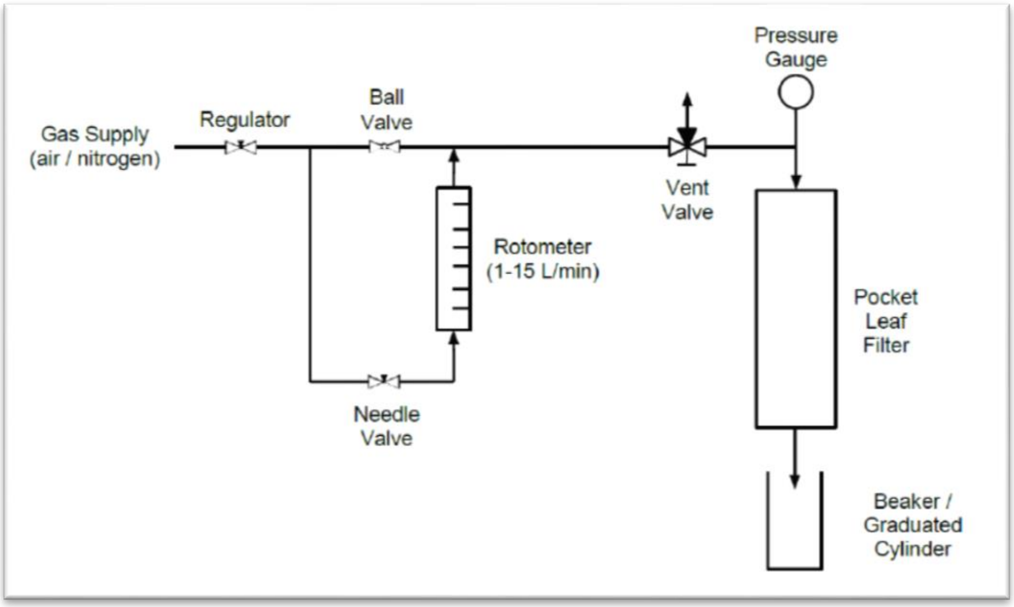


Figure 7: BHS Pocket Leaf Filter for Pressure Testing

Figure 8: BHS Candle Filter Pilot Rental Skid with 1 m^2 of Filtration Area

Figure 9: BHS Candle Filter for Slurry Discharge

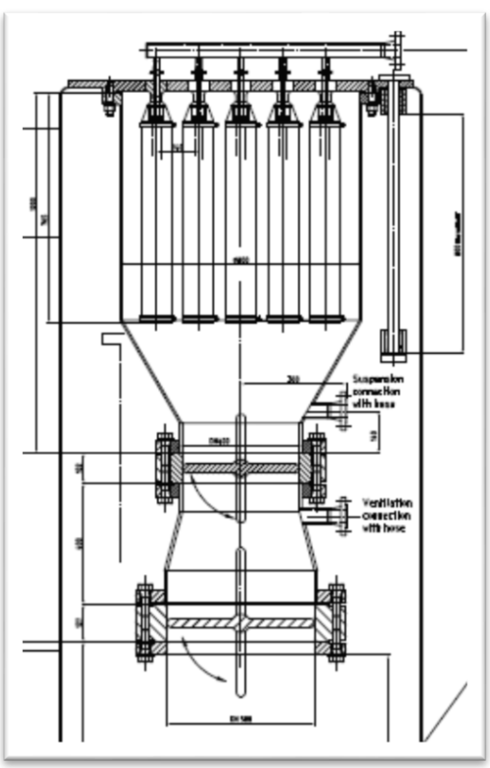


Figure 10: BHS Candle Filter for Pharmaceutical Design with Free-Draining Candles

Figure 11: BHS Pressure Plate Filter including Turnkey Skid