SEPTEMBER 2019 ISSUE 939

The Chemical Engineer

NEWS AND VIEWS FROM THE PROCESS INDUSTRIES, BROUGHT TO YOU BY THE INSTITUTION OF CHEMICAL ENGINEERS

DRUG DISCOVERY

Why developers are turning to performance-enhancing AI

This article first appeared in *The Chemical Engineer*, which is published monthly by the Institution of Chemical Engineers
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Separations under Control

Barry Perlmutter explains developments in containment for eliminating worker exposure during solid-liquid separation operations

IN the 1970s, chemical operations that I have been exposed to used acetone and benzene for the main slurry solid-liquid separation process, as well as for cleaning. In the 1980s, there was a push to minimise solvent use, and research was looking more towards using water as the process liquid, but you still had open filter presses, and rotary drum filters where the entire plant was white from titanium dioxide or pharma stearates.

Fast forward to the present and we all know that processes remain open with filter presses, vacuum filters and centrifuges. Our job these days is to find solid-liquid separation process solutions that can be contained for high solids slurries (greater than 10% solids) during filtration, cake washing and dewatering/drying. The discussion below looks at batch and continuous operations.

FIGURE 1: AGITATED NUTSCHE FILTER-DRYER SHOWING THE OPEN SIDE DISCHARGE VALVE AND THE LOWERED FILTER PLATE FOR ACCESS FOR CLEANING AND CHANGING THE FILTER MEDIA

BATCH OPERATIONS

NUTSCHE FILTER-DRYERS

Nutsche filter-dryers (*see Figure 1*) have been around from the early 1980s and are a mature technology. When containment was required, every chemical engineer wanted to install a filter-dryer. They are sized to take the complete batch from the reactor and process it to completion, meaning final dryness. The agitated nutsche filter-dryer is based upon thick cakes from 5–7 cm up to 30 cm and higher. For this type of filter to be successful, the cake permeability must be able to accept a deep cake without compression. Circular or rectangular filter media with a drainage layer is installed on a perforated filter plate.

The nutsche filter contains an agitator sealed to the vessel by means of a stuffing box or mechanical seal. The agitator, normally three blades, covers the diameter of the vessel. The agitator moves up and down as well as in the clockwise and counter-clockwise directions. The agitated nutsche filter can conduct pressure filtration, cake smoothing, cake washing (displacement and reslurry washing), vacuum and pressure drying, and then automatic cake discharge.

CONTAINED FILTER-PRESSES

Contained filter-presses came onto the market for several reasons. Filter-presses are used everywhere, and when containment became a topic of concern, these manufacturers adapted their technologies. A contained unit did not require a process change and more importantly, these units could operate at a cake thickness down to 2.5 cm, which is not possible in a nutsche filter-dryer. There are several types of contained filter-presses. A typical design is shown in *Figure 2*, where a housing seals the plates.

A CONTAINED UNIT DID NOT REQUIRE A PROCESS CHANGE AND MORE IMPORTANTLY, THESE UNITS COULD OPERATE AT A CAKE THICKNESS DOWN TO 2.5 CM, WHICH IS NOT POSSIBLE IN A NUTSCHE FILTER-DRYER

An improved design (*see Figure 3*), includes pressure filtration up to 1m Pa, cake washing in the forward and reverse direction, cake drying in the forward and reverse direction using pressure blowing and vacuum, as well as automatic cake discharge.

FIGURE 2: ENCLOSED FILTER PRESS SHOWING THE OPEN COVER FOR ACCESS TO THE FILTER PLATES FOR CLEANING AND FILTER MEDIA CHANGING

Circular plates with welded metal or synthetic media are contained in a pressurised housing. This allows for pressure filtration, cakes washing and vacuum or pressure drying. After the cycle is completed, the housing is moved and automatic cake discharge is via scraper knives that move between the plates.

CONTAINED CENTRIFUGES

Contained centrifuges vary in design depending upon the operation and the type of centrifuge such as horizontal peeler, inverting basket, and disk centrifuges. Centrifuges can be blanked or inerted for operation as well as sealed designs. For example, a contained centrifugal disk centrifuge is shown in *Figure 4*.

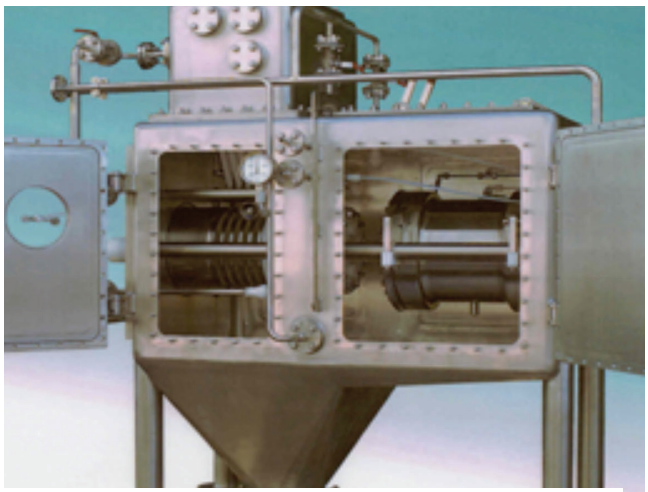

FIGURE 3: CONTAINED FILTER PRESS WITH AUTOMATIC CAKE DISCHARGE SHOWING THE OPEN INTERNAL PRESSURE-RATED HOUSING OVER THE FILTER PLATES FOR CAKE DISCHARGE

FIGURE 4: CONTAINED DISK CENTRIFUGAL SEPARATOR SHOWING THE COMPLETE PRESSURISED ENCLOSURE

CONTINUOUS OPERATIONS

ROTARY PRESSURE FILTER (RPF)

The rotary pressure filter, shown in *Figure 5*, is a continuous pressure filter designed for thin cake to deep cake filtration with cake depths from 6–150 mm. The slowly rotating drum (6–60 rph) is divided into segments (called cells) each with their own filter media (synthetic cloth or single or multilayer metal) and outlet for filtrate or gas. The outlets are manifolded internally to a service/control head where each stream

FIGURE 5: ROTARY PRESSURE FILTER SHOWING A FULLY-CONTAINED HOUSING INCLUDING A PRESSURISED CAKE DISCHARGE CHUTE AND PRESSURISED FILTRATE AND GAS OUTLET NOZZLES

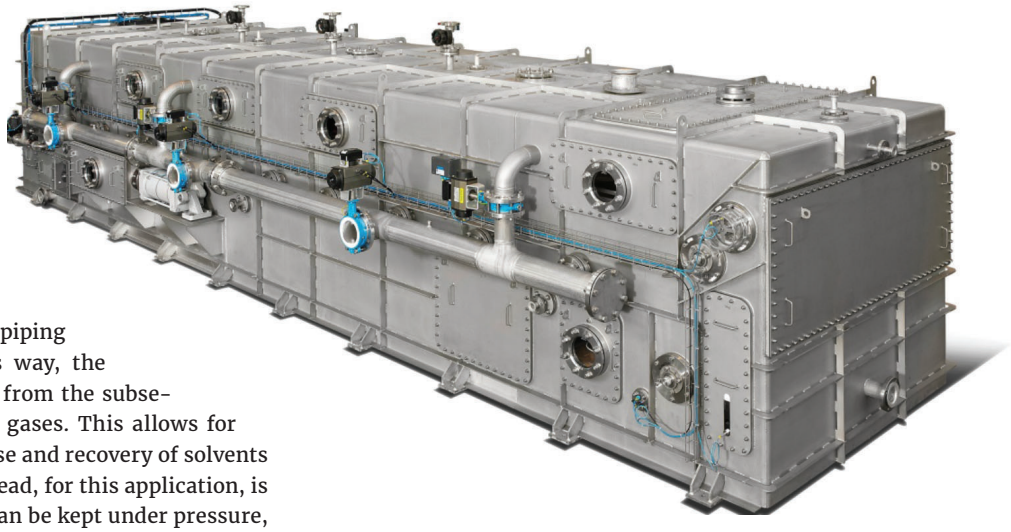

FIGURE 7: CONTINUOUS-INDEXING VACUUM BELT FILTER SHOWING FULLY-CONTAINED PRESSURISED HOUSING WITH ALL MAINTENANCE POINTS ON THE EXTERIOR OF THE HOUSING

can be directed to a specific plant piping scheme or collection tank. In this way, the mother liquor can be kept separate from the subsequent washing filtrates and drying gases. This allows for better process control as well as reuse and recovery of solvents and the gases. The service/control head, for this application, is pressure rated so the liquefied gas can be kept under pressure, acting as a liquid. Cake washing by displacement as well as gas dewatering follows filtration and continues to automatic cake discharge via a scraper knife or gas blowback.

PRESSURISED VACUUM DRUM FILTER (P-VDF)

Another alternative to the RPF is the pressurised drum filter (P-VDF), *see Figure 6*, which is a rotating drum inside a pressure vessel. The unit consists of a filter drum, slurry trough, agitator, wash bars and a pressure let-down rotary valve. The process begins by closing the pressure vessel, pressurising the vessel with compressed gas. The rotary valve is also pressurised for sealing, and the filter trough is filled via the suspension feed pipe. The agitator is started to keep the solids in suspension. Filtration, cake washing and drying are by vacuum operation.

INDEXING VACUUM BELT FILTERS

The continuous-indexing vacuum belt filter provides for vacuum filtration, cake washing, pressing, and drying of high solids slurries. The technology is based upon fixed vacuum trays, a continuously-feeding slurry system and indexing or step-wise movement of the filter media. In practical terms, the operational features of the belt filter can be viewed as a series of Buchner funnels.

For the process operation, due to the stepwise operation of the belt, washing and drying efficiencies are maximised with the stopped belt and a plug-flow mechanism for gases and liquids. Cake pressing and squeezing further enhances drying. Finally, the fixed trays allow for the mother liquor and the wash filtrates to be recovered individually and recirculated, recovered or reused for a more efficient operation. Steaming and counter-current washing can be integrated into the design. *Figure 7* shows the contained design.

FIGURE 6: PRESSURE-VACUUM DRUM FILTER SHOWING AN OPEN HOUSING FOR ACCESS TO THE VACUUM DRUM FOR INSPECTIONS AND MAINTENANCE

FINAL THOUGHTS

Process engineers have many choices to contain an operation, but it is not an easy choice. First, is the process batch or continuous? Is it a thin-cake or thick-cake operation? What is the filter media (synthetic or metal)? What are the critical process steps? What about maintenance and other parameters?

As you can see, the design questions go on and on. The decision tree for a contained process is the subject of another discussion. In the end, whatever you decide, please be sure that you involve process, production, operations and maintenance in your decisions. You can follow my blog at www.perlmutterunfiltered.com or my "Handbook of Solid-Liquid Filtration" (https://bit.ly/2Ml2xER) for further insights. ■

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