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CATALYST RECOVERY FROM CONTINUOUS FLOW REACTORS WITH MOTT HYPULSE[®] LSM FILTERS

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ABSTRACT

The Mott HyPulse® LSM filter recovers fine, suspended catalyst from continuous flow, stirred tank reactor (CSTR) systems with a single filter unit. Traditionally, multiple dead-end filters with holding tanks have been required to maintain constant product flow. The all-metal design of the LSM filter permits operation at reactor temperature and pressure. By operating the LSM in a recirculating loop around the reactor, the catalyst is always kept at reaction conditions which minimizes activity loss due to depressurization and mechanical stress. The filter cake is controlled dynamically with the slurry recirculation rate, assisted by on-flow reverse backpulsing.

The paper describes LSM filtration technology, gives operating characteristics based on pilot and full scale experiences, and lists selection criteria for this novel filter.

INTRODUCTION

In the past, continuous stirred tank reactors with solid catalyst particles utilized in-tank candle filters to retain the catalyst in the reactor. Reaction product is withdrawn through the candles which are usually covered with either felt or cloth filter socks, that would be backpulsed periodically to remove the catalyst cake. Unfortunately, these filter media socks would abrade apart quite frequently (typical sock life is one month), which would require reactor shutdown, candle removal, and redressing with new socks.

In addition, the socks were an open media that allowed fine attrited catalyst particles to pass through, which required costly downstream filter aid or cartridge filtration, not to mention the loss of expensive catalyst. Attempts at using tighter filter media usually failed because of either blinding or the tighter media would attrite faster. As a consequence, a new more robust filtration system had to be developed, and the Mott HyPulse® LSM system based on porous sintered metal media fulfilled the requirements. This paper describes this new filter technology and details one typical application from laboratory testing to piloting to full-scale plant production.

APPLICATION DATA

The application described in this paper concerns the retention of fine catalyst particles from a continuous flow, stirred tank reactor (CSTR) where an organic intermediate is isomerized at 60°C temperature and up to 90 psig pressure. The reactor effluent contains 1.5 to 10 wt% of suspended catalyst ranging in size from 0.5 to 100 micron and must be filtered to less than 1 ppm catalyst at a maximum differential pressure of 12 psi. After filtration the recovered catalyst is to be recycled to the CSTR. Completely enclosed operation is desired to prevent catalyst de-activation as well as product contamination.

High efficiency catalyst retention is an important objective because it minimizes the possibility of isomer loss due to reverse catalytic reactions in downstream operations. In addition, low solids content in the product reduces wear and tear in these operations, aside from savings on catalyst replacement costs. Switching from felt or cloth type filter socks to porous metal media increases reactor availability and eliminates maintenance costs associated with the sock change-outs.

Mott porous metal media made from precision sintered stainless steel powder was selected for this application for several reasons: First, due to highly uniform porosity and permeability, the media can retain catalyst particles down to the 0.5 micron range at moderate pressure drops without by-passing; secondly, the mechanical strength of the rigid powder matrix allows in-place cleaning by reverse flow over thousands of filtration cycles; and lastly, the media is chemically compatible with the fluid components.

LABORATORY TESTING

Basic filtration data was obtained with 4 wt% slurry samples in small scale filters at the Mott Corporation laboratory. Dead-end filter testing with a Mott 2 micron grade 70 mm disc filter assembly showed feasibility for repeated cycles with regards to filtrate quality, throughput and filter cake release during backpulsing. Filtrate flux rates of 0.25 to 0.4 gpm/ft² at terminal pressures of less than 5 psi could be maintained during 30 to 45 minute long cycles. The 2 micron grade media showed initial particulate breakthrough measured at 1.5 NTU turbidity. The composite filtrate sample turbidity, however, was lower at 0.5 NTU. This turbidity was correlated to 2.4 ppm and 0.45 ppm of total suspended solids (TSS).

A laboratory scale crossflow filter with 0.087 ft² area equipped with a Mott 1 (one) micron grade media was evaluated for continuous recirculation filtration as an alternate to dead-end filtration. The finer micron grade produced filtrate at 0.3 gpm/ft² flux with less turbidity than the 2 micron grade disc filter (i.e. 0.25 NTU = 0.21 TSS), but at a higher pressure drop. The crossflow filter, due to the continuous cake thinning action of a 10 ft./sec. axial mainstream flow, developed a stable pressure drop and did not indicate a need for media backpulsing.

FILTER SELECTION

Based on the laboratory results, DuPont decided to recover the catalyst outside of the reactor with either dead-end or crossflow-type filters in order to segregate reaction from separation process conditions. The three filter options that were considered are described next.

The first option LSI filter is a dead-end type filter with single open-ended 2.5" diameter x 60" long porous metal elements mounted standing up in a cylindrical housing. The elements are fed with slurry from the bottom inlet and filter from the inside to the outside with solids being retained as a cake on the inner surface of the filter media. This filter is operated in batch mode with a cake discharge step between filtration cycles. For continuous process filtration, dual filters are piped in parallel and operated alternately. The intermittently recycled catalyst is returned to the CSTR via a feed tank to control the catalyst inventory in the reactor. A flow sheet for the LSI filter option is shown in Figure 1.

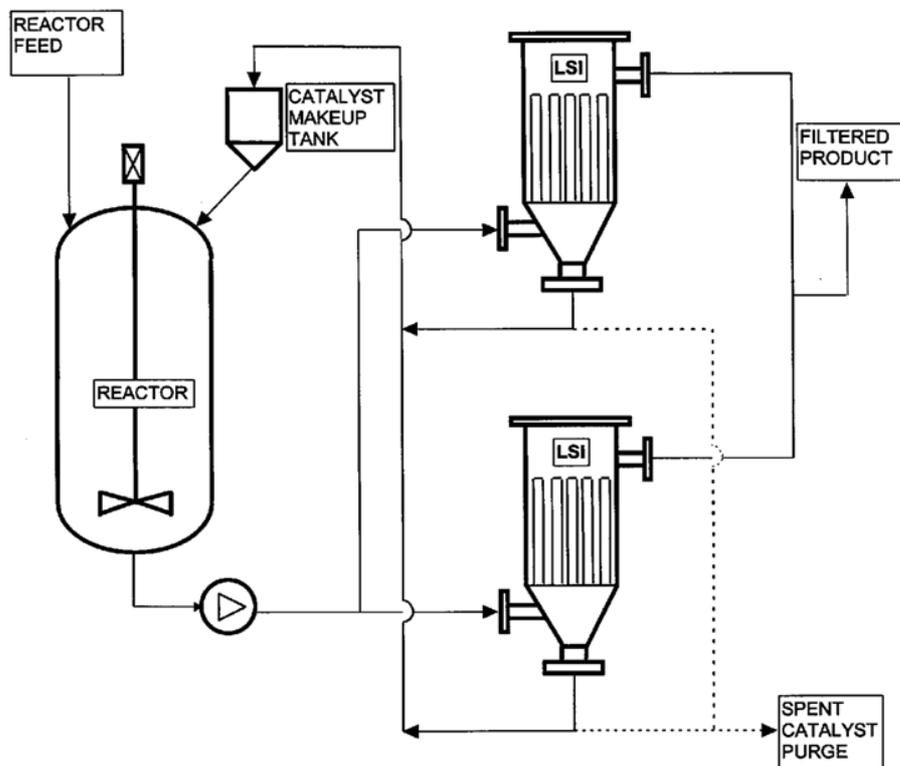


Figure 1: LSI Filter Option Flowsheet

The second option LSX filter employs 1/2" diameter porous metal filter tubes and is operated in the crossflow mode for continuous filtrate flow. The reaction slurry is circulated in a loop from the CSTR through the LSX and back. By maintaining high axial slurry velocity in the filter tubes, the filter cake is minimized for constant flow resistance. The flow diagram for option LSX is shown in Figure 2.

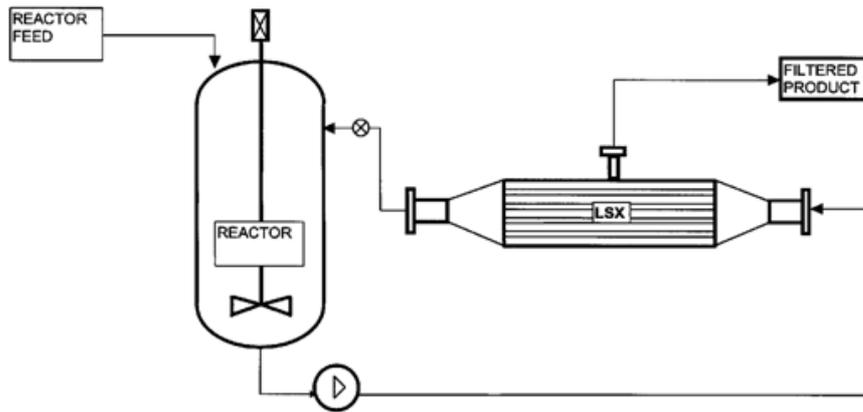


Figure 2: LSX Filter Option Flowsheet

The third option, the patented LSM filter (see Ref [1]) with double open-ended filter elements is a hybrid between LSI and LSX filters. Operating vertically, the feed slurry is fed from the top into the filter elements where filtrate is withdrawn in radial outward flow. A portion of the feed flow is recirculated from the bottom outlet to the reactor (see Figure 3). The driving pressure for filtration is provided by a back pressure control valve on the slurry recycle line.

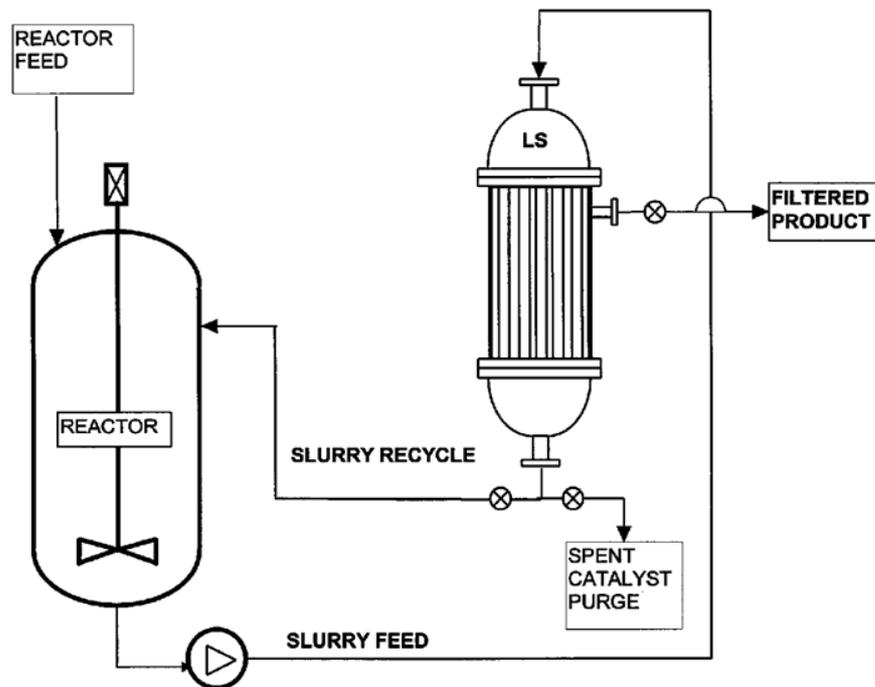


Figure 3: LSM Filter Option Flowsheet

Cost estimates for full size filters based on laboratory test data and operating cost projections were used to select a filter (see Table 1). The traditional barrier type LSI filter option, although with lowest cost per unit area, requires dual filters and a catalyst recycle tank which increases its capital costs far beyond the LSX and LSM options. Capital costs for the LSX options are in the mid-range, but high

pumping costs, concern about catalyst degradation, and the non-availability of catalyst purging offset the viability of this option based upon media cleaning. Low initial capital costs and low operating costs (energy and catalyst) give the LSM filter the highest score, if all issues are weighed equally. Though catalyst degradation and cleaning requirements of the LSM option are potentially superior to the other options, pilot testing was required to validate these benefits.

Filter System	LSI	LSX	LSM
	Dual Barrier	Single Crossflow	Single Recirculating
Mode of Operation	Alternate Batch	Continuous	Continuous
Capital Costs	1	3	5
Energy Costs	5	1	3
Catalyst Inventory	1	5	5
Catalyst Degradation	3	1	3-5
Media Cleaning Requirements	2	5	1-5
Spent Catalyst Purging From System	5	1	5
TOTAL	17	16	22-28

Scale: 1 - Worst 5 - Best (Total assumes equal weighing of all issues)

Table 1: Comparison of Filter Options

PILOT TESTING

Single element testing at a DuPont pilot plant validated the LSM filter concept under actual production conditions. A 0.5 micron grade media was evaluated since 1 and 2 micron grade media had shown initial solids breakthrough. Filtrate quality was consistently below 1 ppm TSS as required for the process upgrade.

The pilot filter demonstrated freedom of media plugging over extensive test periods. In-place media cleaning was infrequent as depicted in Figure 4 which shows filter pressure drop over 700 hours of continuous operation. It was observed that the downward slurry flow frequently washed off excessive cake buildup without operator intervention. For media cleaning, the filtrate was shut off momentarily, followed by reverse flow filtrate pulsing. During the short pulse, the back pressure valve at the bottom outlet was fully opened to flush solids out of the filter.

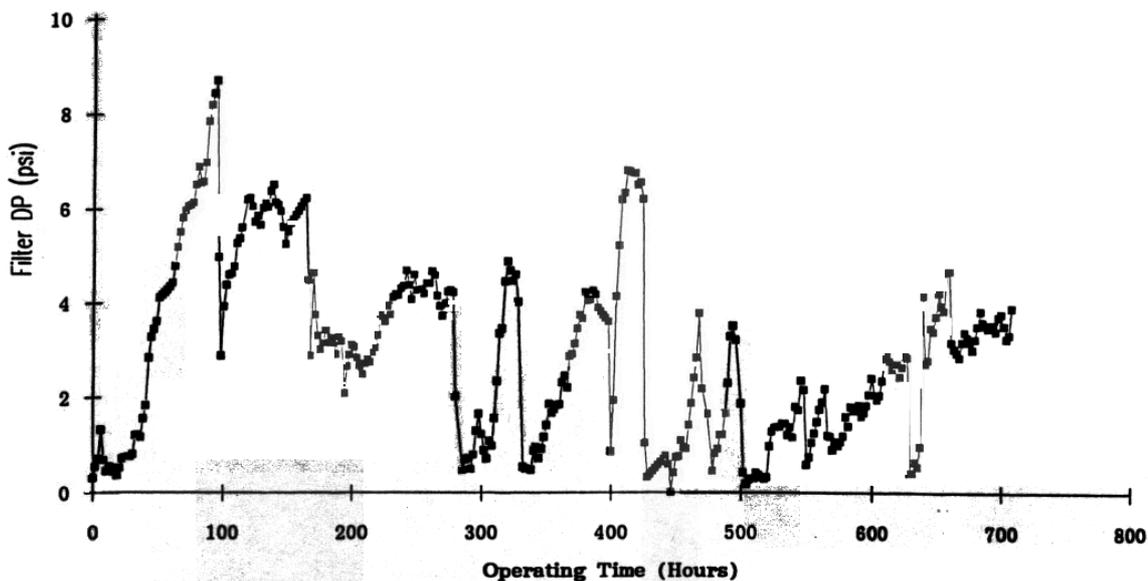


Figure 4: Pressure Drop Across Filter During Pilot Testing

When the axial velocity of the feed slurry entering the 1.5" diameter element was reduced by 25%, the steady-state pressure drop increased by 50% to 10 psi (see Figure 5). Further reductions in slurry feed rate caused back-up of catalyst on the back pressure valve.

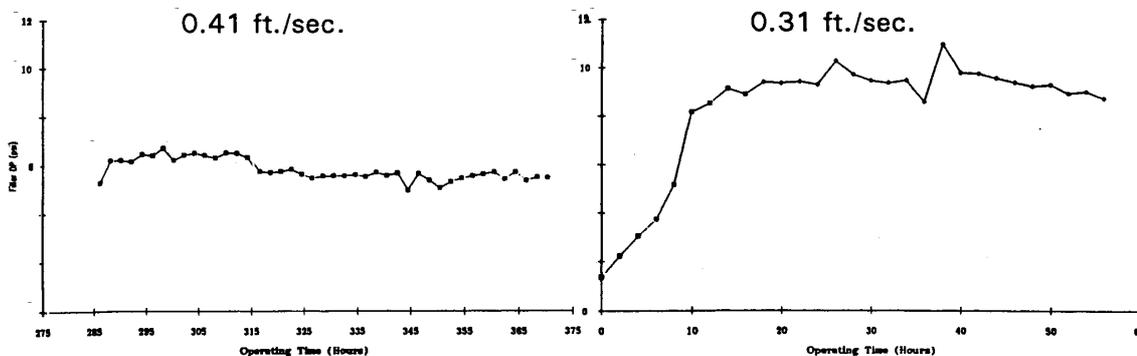


Figure 5: Steady-state Pressure Drop At 0.41 and 0.31 ft./sec. Axial Flow Rate

To purge spent catalyst from the reactor system, the LSM filter is operated as a dead-end filter without recirculation until the filter is loaded. The filter cake is then discharged into a spent catalyst receiver (see Figure 3). Top-feed LSM filter operation in the dead-end mode is also recommended for once-through type catalysts with high settling velocities. Downward flow in the LSM filter elements eliminates size classification problems common to upward flow filters.

SCALE-UP

The full scale filter unit comprised 91 elements, each with 1.5" OD x 0.047" wall x 100" long 316L SS media in 0.5 micron grade porosity, mounted in a 24" diameter filter housing (see Table 2). The elements were welded into the tubesheet to eliminate potential solid bypassing through deteriorating polymeric O-ring seals between each element and the tubesheets (see Figure 7). By selecting 100" long elements, both housing size and recirculation rate were minimized. The recirculation rate was initially set at 0.4 ft./sec. axial velocity, but could later be reduced to 0.3 ft./sec. Operation of the reactor/filter aggregate was fully automated with throughput controlled with the filtrate outlet valve via reactor level control; filter pressure drop was limited to 12 psi by on-flow backpulsing.



Figure 7: All-welded filter bundle for 24" diameter LSM filter (shown horizontally - top tube sheet on right)

Scaling up the filter area from the pilot test by a factor of 1000 was successful due to the similarity in pressure and flow conditions between both filter sizes. Since the inside-out flow path in the LSM filter eliminates interactions among the parallel operating elements, increasing the number of elements was not problematic.

	PILOT	FULL-SCALE
Element Size	1.5" OD x 14" Long	1.5" OD x 100" Long
No. of Elements	1	91
Housing Size	2"	24"
Filtrate Quality	< 1 PPM	< 0.01 PPM
Maximum Pressure Drop	8 - 12 PSID	8 - 12 PSID

TABLE 2: LSM Filter Scale Up

SUMMARY

Since start-up in July of 1994 the fully automated LSM filter has been operating on a 8,000 hr/year production schedule with the original filter elements. Pressure drop has been stable and filtrate quality has been maintained at lower solids content that experienced during pilot testing.

The plant has eliminated labor and replacement costs for primary filter socks as well as final filters. Product quality has increased and catalyst life was extended significantly. These savings recovered the filter capital costs during the first year of operation.

References

- [1] U.S. Patent No. 4,552,669 (Nov. 12, 1985), R. Sekellick, assigned to Mott Metallurgical Corporation