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Relationship, Selection, and Optimization of Filter Aid, Filter Media and Clarification Technologies for Contaminant Fines Removal from Process Slurries and Liquids

INTRODUCTION

In many chemical processes there is the need to remove fine solid contaminants less than 5 microns (µm), from process liquids. When the suspended solids content is dilute, typically less than 1% by weight (wt. %), the removal process is called clarification or polishing filtration to produce a clean liquid product. Typical applications include catalyst recovery, polymer and resins, active pharmaceutical ingredients, removal of unreacted chemicals and byproducts, polishing of grey water, etc.

Often in polishing applications the solids are very fine or amorphous which can be difficult to filter. When filtered, the solids will create a thin coating over the filter media and immediately reduce the filtration rate to an unacceptable level.

In these difficult filtering cases, pretreatment can be used to improve filtration properties and efficiently remove the fine solids from the process liquids. Chemical or physical pretreatment, such as use of flocculants, is one option that can be used to effectively change the nature of the suspended solids. Alternatively, a better filtering medium can be added to the suspension to increase the permeability of the cake. This medium is known as filter aid. An efficient and economical filter aid must have the following properties (World Minerals, 1999):

1. Have rigid, complex shaped, discrete particles
2. Form a permeable, stable, incompressible filter cake
3. Remove fine solids at high flow rates
4. Be chemically inert and insoluble in the process liquid

This paper discusses the use and selection of filter aid and filter media in conjunction with clarification technologies for the removal of fines from chemical process liquids. This article continues with case studies performed to determine the ideal ratios of filter aid for body feed to the suspended solids content as well as to the particle size distribution of these fines.

TYPES OF FILTER AID

Diatomite, perlite, and cellulose are the most frequently used filter aid materials in filtration. Other organic material such as potato starch particles and rice hull ash are also used in the industry but are less common (Sulpizio, 1999).

Diatomite

Diatomite, also known as diatomaceous earth (DE), is comprised of the microscopic silica skeletons of prehistoric plants. These skeletons are called diatoms. This material is mined and calcined to destroy any organic matter. The diatomaceous earth is then milled and separated into various filter aid grades through the use of air flotation (McKetta & Cunningham, 1976). The final product is a soft, powdery material resembling chalk.

Diatomite filter aids are advantageous due to their rigid and highly porous structure. When deposited on a filter media the diatomaceous earth forms a non-compressible cake and acts as a sieve to remove fine solids. The micropores in the diatoms allow flow through the particle while the fine solids are captured between the diatom granules. Being comprised of mostly silica, diatomaceous earth is inert in a wide range of applications.

MARINE DIATOMITE

Perlite

Perlite is formed from glasslike volcanic rock that consists of small particles with cracks that retain water and gas. The perlite is mined and transformed into filter aid by heating the ore to the melting temperature. Once the ore reaches the melting point, the molten volcanic glass expands and fractures due to the emission of the steam and gas. This expanded material is then crushed and classified to provide different filter aid grades.

Compared to diatomaceous earth, perlite has a lower specific weight which allows less filter aid to be used. The primary advantage of using perlite compared to diatomaceous earth is the relative purity. Diatomite may contaminate process liquids with dissolved salts and colloidal clays that are inherent to the diatomaceous earth (Cheremisinoff, 1998). Perlite does not share this risk.

EXPANDED MILLED PERLITE

Cellulose

Cellulose is used as filter aid to a lesser degree than diatomaceous earth and perlite. This is due to its higher cost and lower filtration efficiency. There are some advantages to the use of cellulosic filter aid versus diatomite or perlite. Cellulose is combustible and is useful in the recovery of valuble metals. Cellulose is also compatible with hot caustic solutions where diatomacous earth and perlite are not (Cheremisinoff, 1998).

S.E.M. Images of Diatomite, Perlite, and Cellulose Filter Aids (Sulpizio, 1999)

GRADES OF FILTER AID

For each type of filter aid there are different grades of material which vary in particle size. Typical filter aid particle size ranges from 5µm to 100µm. As filter aid particle size decreases, the ability to capture fine solids increases, thus the filtrate clarity improves. Also as the filter aid particle size decreases, the cake resistance increases. Therefore the finer the filter aid, the throughput per unit area decreases. Given this relationship it is necessary to balance filtrate clarity with filtration rate. It is important to use the largest filter aid grade possible while still achieving the filtrate clarity target.

Since the solids of every application are different, there is no rule for matching filter aid grade to suspended particle size. Instead it is recommended to run benchtop scale filtration tests with various filter aid grades. The rate of filtration can be measured and filtrate clarity observed which will help to select the optimal filter aid product.

FILTER MEDIA

Filter media is the septum on which the filter aid solids are retained while the clear liquid passes through. Most often the media is constructed of a woven synthetic polymer or alloys. The filter media is supported by a rigid supporting structure which is also porous to allow liquid to travel through.

In precoat and body feed applications the filter media pore size should be approximately equal to the median particle size of the filter aid. The exact filter media pore size is not critical so long as some of the filter aid is retained. Once the filter aid begins to build on the woven filter media, the cake becomes the new primary surface for particle capture. Selecting a filter media with too large of a pore size will result in a long turbid cycle as the bridging of the filter aid will take a long time and may be unstable. Selecting a media that has too small of a pore size will result in a reduced filtration flux rate due to the added resistance.

FILTER AID UTILIZATION METHODS

Precoat

In the precoat method, filter aid is used to generate a thin layer of solids on top of the filter media. Once formed, the filter aid cake functions as the primary filter media. Therefore, the filter cloth is no longer the real filter when precoat is used. For that reason, the filter cloth should be as open as possible while still retaining the filter aid material.

The precoat process is achieved by mixing the filter aid into clear liquid or mother filtrate in a precoat tank. A precoat slurry concentration in the 0.3 – 0.6wt% has been found to work best for diatomite filter aids (McKetta & Cunningham, 1976). This slurry is then recirculated through the filter where the solids are captured by the filter media. The clean filtrate is recirculated back into the precoat tank (Figure 1). The precoat thickness should be thick enough to ensure that the entire media surface is coated but thin enough so that it does not provide significant resistance to filtration. In order to prevent the precoat from settling in the pressure vessel and creating an uneven precoat, a feed flow rate of 1 GPM/ft^2 is ideal. Typical precoat thicknesses are in the 1-3mm range for polishing filtration (Svarovsky, 1990).

There are several advantages to using precoat in a clarification processes. Use of a precoat helps prevent the filter media from becoming clogged by the slurry fines, thus extending filter media life. This is especially critical when filtering amorphous or slimy solids. Precoating helps provide immediate filtrate clarity. The precoat layer acts as a depth filtration bed which will help trap fine particles that would typically pass through the filter media. Precoat helps filter cake solids discharge from the filter media at the end of a filtration cycle by providing a cleavage plane from the filter media (World Minerals, 1999).

Body Feed

In body feed filtration applications, the filter aid is blended with the slurry feed either by dosing concentrated filter aid suspension into the slurry feed and in-line mixing (Figure 1), or by mixing the filter aid into the entire slurry batch and maintaining agitation.

The filter aid solids and the native solids continuously build a filter cake until the maximum cake height is reached or the maximum operating pressure is reached. By adding filter aid into the slurry feed, the resulting filter cake is more porous, allowing higher and longer sustained flow rates. Body feed also helps to restrict solids movement which improves filtrate clarity.

The goal of the optimal body feed concentration is to maximize the flow rate of a filtration cycle while avoiding feeding too much filter aid solids. Using too much body feed will cause the filter to reach the maximum filter cake height faster than necessary. When using a body feed without precoat, there can be an initial turbid filtrate while the cake begins to build on the filter media. This initial turbid bypass will generally be much shorter than what would be observed filtering the raw slurry. It is possible to recycle this turbid filtrate back to the slurry feed tank to verify no solids bypass occurs.

Filter Aid Application Summary

	Typical Filter Aid Applications
Filter Media Only	• Crystalline, easy to filter solids • Low solids concentration • Particle size can be matched to cloth pore size • Solids or filtrate is the product
Media & Precoat	• No slimy or amorphous solids • Retention of fines is critical • Solid particles are fine enough to penetrate the precoat bed without blinding precoat OR • Solid particles are coarse enough to deposit on top of precoat without blinding • Filtrate is the product
Media & Body Feed	• Solids are not slimy • Immediate retention of fines is not critical • Solids are difficult to filter
Media & Precoat & Body Feed	• Solids are slimy or amorphous and difficult to filter • Immediate fines retention

UTILIZATION OF FILTER AID FOR CLARIFICATION APPLICATIONS

Filter aid is utilized in clarification filtration processes using two methods: precoat and/or body feed addition. These two methods can be used individually or they can be combined to gain the benefits of both methods. Various filters can utilize filter aid in their filtration cycles. Candle Filter and Pressure Plate Filter are two filtration technologies and are described below. Other filters that can utilize filter aid are filter presses, pressure leaf filters and Nutsche filters.

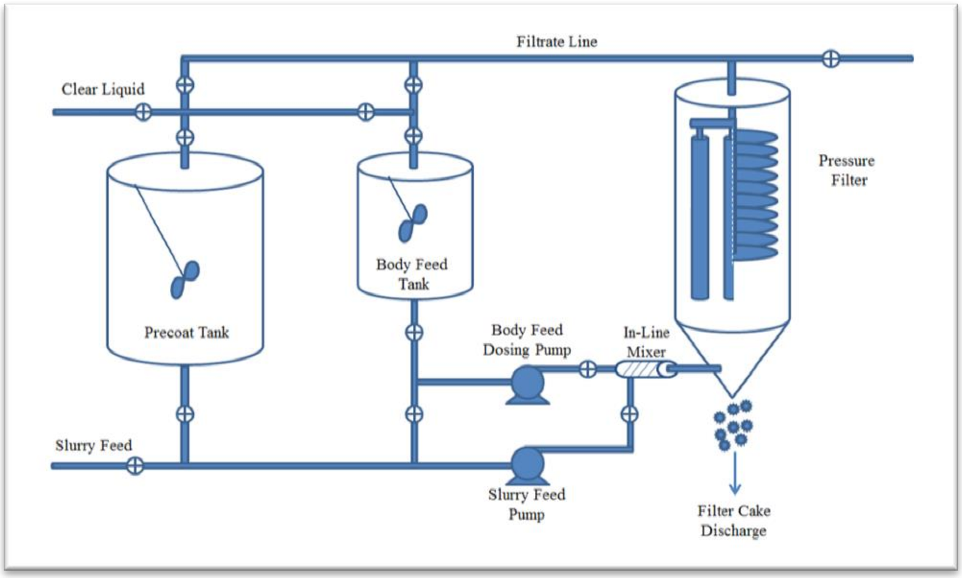

Figure 1: Process flow diagram for precoat and body feed processes with filter aid and filtration via candle or plate filter (Sentmanat, 2011)

Candle Filter Technology

The candle filter is a batch-operated, pressure driven filter. The pressure vessel is filled with tubular filter elements called filter candles. A filter candle is comprised of a dip pipe for the filtrate, a perforated core with supporting tie rods and a filter sock. Each of these candles is installed on horizontal manifolds called registers. Depending on filter size, each register may contain 1 – 20 candles. Given the staggered arrangement and size of the candles on the register, a large filtration area can be provided in a small vessel (Perlmutter, 2015). This arrangement is particularly advantageous in low solids applications <1wt% both with and without filter aid. The large filtration area leads to high throughputs while the cake slowly builds to the maximum filter cake height.

During operation a feed pump forces slurry through a flange into the bottom of the pressure vessel. The solids build up on the outside of the filter sock while the filtrate flows into the candle, through the dip tube and registers. The process continues until the maximum pressure drop, cake thickness, minimum flow rate, or maximum filtration time is reached.

The cake is dewatered by draining the vessel and pressurizing with compressed gas. During cake discharge, the filter socks are inflated by low pressure gas. The process breaks apart the dry cake which falls from the candles and through the discharge valve at the bottom of the vessel.

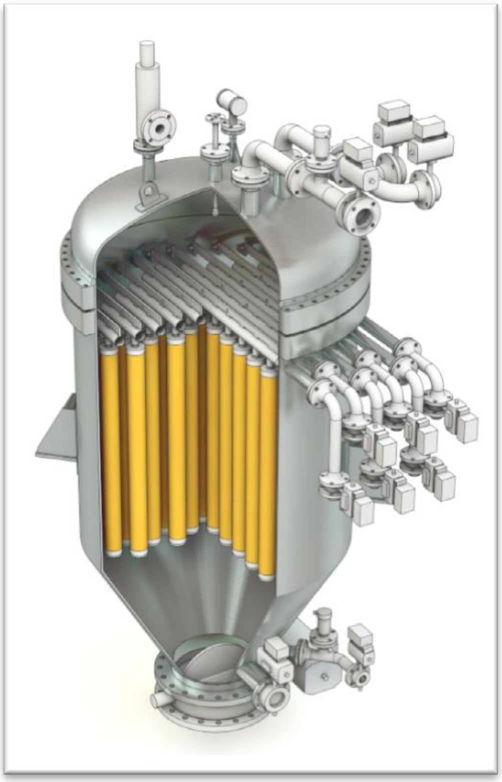

Figure 2: BHS Candle Filter Technology (BHS-Sonthofen, 2017)

Pressure Plate Filter Technology

Similar to the candle filter, pressure plate filters are comprised of filter plates contained within a pressure vessel. Operation is essentially the same as the candle filter with a pump driving slurry into the filter. Solids are captured on the filter media surface while the filtrate is driven away from the vessel.

The main difference between the two technologies is the orientation of the filtration surface and cake discharge mechanism. Instead of vertically oriented candles, the plate filter contains slightly sloped, horizontally oriented, conical-shaped filter plates. The plates have filter media sealed on the top surface of each plate. An opening in the center of the plates allows the filtrate to travel between the plates and out of the vessel. Cake discharge is done by a back pulse of gas in combination with vibration of unbalanced motors. Other cake discharge techniques include spinning plates or manual discharge where the plate stack is removed from the vessel.

Due to the horizontal orientation of the plates, the pressure plate filter is well suited for processes that require a high degree of versatility and cake stability such as incorporating a precoat or cake wash stage. With the vertical orientation of the candle filter, disruption in the positive pressure of the vessel can result in the cake falling prematurely from the media due to gravity. With the horizontal orientation of the filter plates, the cake would be contained on top of the plates if any pressure disruption were to occur. Just like the candle filter, through the use of BHS designed piping and valves the process can be fully automated which mitigates this risk.

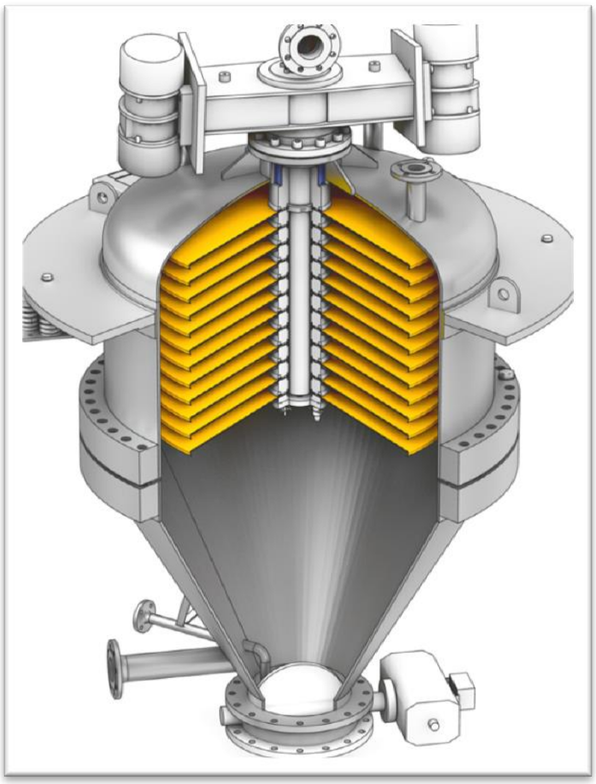

Figure 3: BHS Pressure Plate Filter Technology (BHS-Sonthofen, 2017)

BENCHTOP SCALE STUDY:
OPTIMIZING BODY FEED CONCENTRATIONS IN RELATION TO SUSPENDED SOLIDS CONTENT IN UNTREATED SLURRY

The following study discusses and illustrates a method to physically test and determine the optimal filter aid concentration for a given filter aid grade, filter media, solids size and concentration.

The goal of the case study performed by BHS is to determine the optimal starting point for body feed concentration for suspensions. The tests incorporated slurries comprised of corn starch in water and bentonite clay in water.

Test Setup and Apparatus

The test setup was arranged as shown in Figure 4. A BHS Pocket Leaf Filter (PLF) containing a 12 micron filter media was connected by braided tubing to an agitated slurry tank capable of holding pressure. Hyflo grade diatomaceous earth was utilized as the body feed filter aid for all tests (Table 1).

Median Particle Size	30.1µm
Median Pore Size	7.0 µm
Permeability	1.10 D'Arcys
150 Mesh (% Retained)	7.0 %

Table 1: Hyflo Super-Cel DE Filter Aid (World Minerals, 1999)

The BHS Pocket Leaf Filter apparatus is used for benchtop scale pressure and vacuum filtration tests, to determine flux rates, filtrate clarity, filter media, cake thickness, washing and drying efficiencies, cycle times, performance versus quality parameters and qualitative cake discharge. The PLF is jacketed for heating or cooling and is rated 90 psig to full vacuum. The BHS PLF has a 20cm^2 of active filter area and a filling capacity of 400mL.

The filter medium used for the test series was a multifilament PEEK cloth with a 12µm pore size produced by SEFAR; part number 17-2005-SK012. This filter media was selected due to its high air permeability (150 L/m^{2}s) and small pore size. Due to the high air permeability the resistance from the filter medium is minimized allowing better analysis of the cake resistance. Additionally with a 12µm pore size and an average filter aid particle size of 30µm, the turbid bypass at the start of the filtration cycle is minimized which helps the repeatability of the experiment. In a full scale application, a filter media with a pore size of up to twice the average filter aid particle size could be an appropriate selection.

Batches of slurry were produced by adding measured amounts of corn starch or bentonite into a beaker and mixing with water. Filter aid was added at various concentrations by weight to the suspension and allowed to mix for fifteen minutes. The entire suspension was added to the agitated slurry feed tank and used to pre-fill the BHS pocket leaf filter.

The agitated pressure vessel was used to continuously mix suspended solids and filter aid during the test runs. This vessel was connected to the BHS pocket leaf filter via braided tubing. The pocket leaf filter contained a 12μm woven filter media. Filtration was driven by using 30psig compressed air to pressurize the slurry feed tank which then fed into the pocket leaf filter. After applying pressure to begin filtration, the filtrate mass was measured and recorded in respect to time.

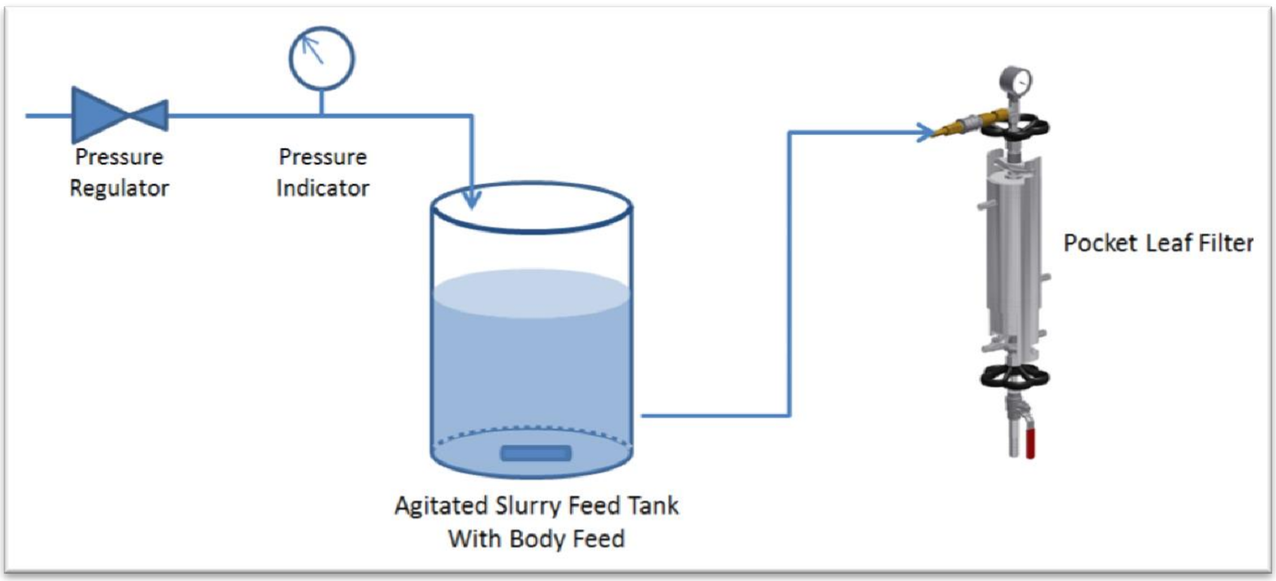

Figure 4: Filtration test setup diagram

Test Results

Corn starch was mixed with ambient temperature water at a concentration of 1wt% suspended solids. Hyflo diatomaceous earth (DE) filter aid was mixed into the suspension at various concentrations ranging from 0wt% to 2wt%. The suspension was filtered at 30psig and filtrate mass recorded versus time. In general, as the body feed concentration increased, so did the filtration flux rate (Figure 5).

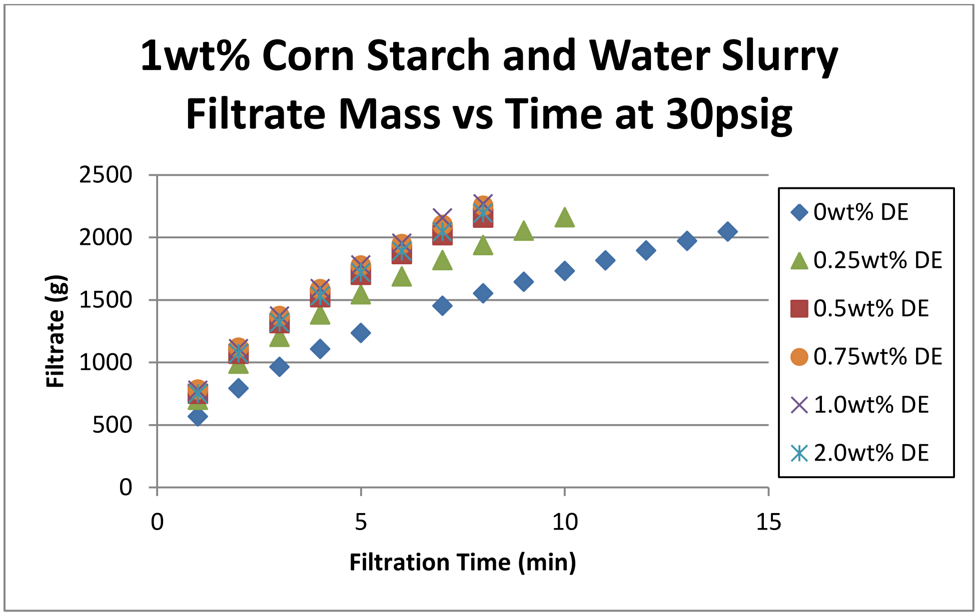

Figure 5: Filtration results for corn starch and water filtration with Hyflo DE body feed at various concentrations

For each test run and data point, filtration flux rate was calculated using the following formula (Equation 1):

$$X(t_{sec})\frac{kg_{filtrate}}{m^2 * s^{0.5}} = \frac{m_{filtrate,t}}{A_{PLF} * \sqrt{t_{sec}}} \quad \text{(Equation 1)}$$

$X(t)$ = Filtration flux at time (t_{sec})
$m_{filtrate}$ = Mass of filtrate at time (t_{sec})
A_{PLF} = Filter area of PLF test unit ($0.002m^2$)

A plot of the average flux rates for each test at varying body feed concentrations, indicates that the optimal body feed concentration is equal to the original suspended solids content in the slurry.

- In Figure 6, the suspended solids concentration is 1wt%. The highest filtration flux is achieved when the body feed concentration is also 1wt%.
- In Figure 7, the corn starch concentration content in the slurry is 0.5wt%. The highest filtration flux is achieved when the body feed concentration is also 0.5 wt%.

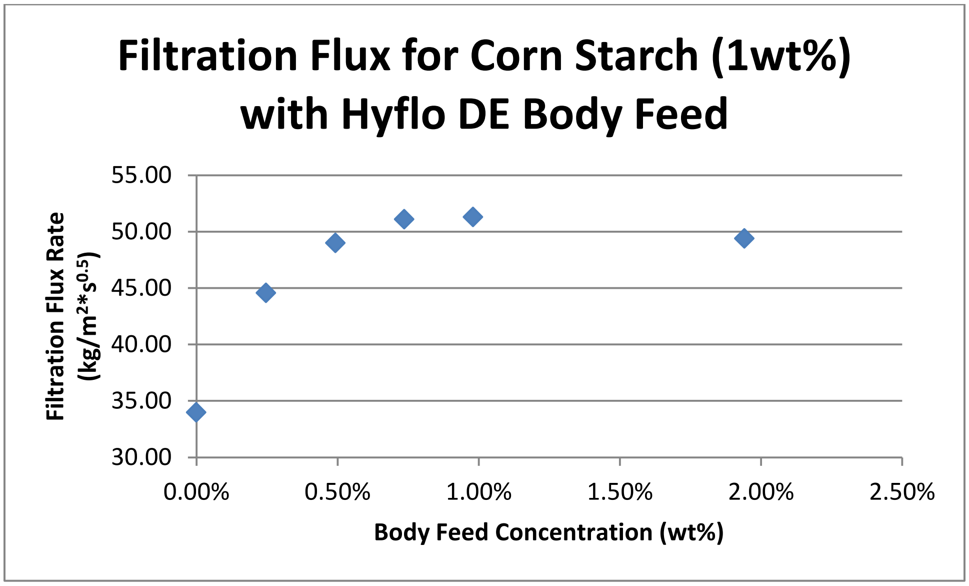

Figure 6: Average filtration flux rate for corn starch (1 wt.%) with various concentrations of Hyflo DE at 30 psig filtration pressure

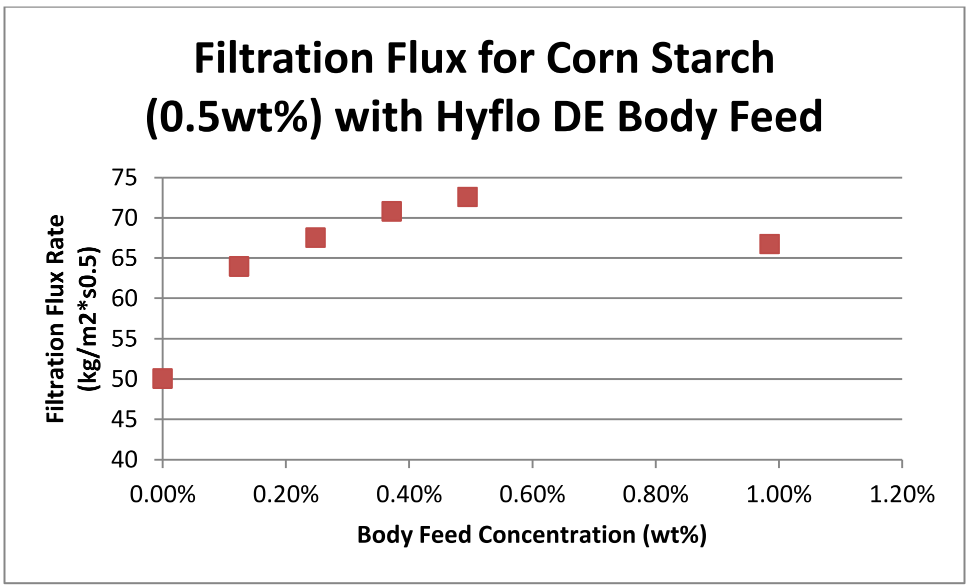

Figure 7: Average filtration flux rate for corn starch (0.5wt%) with various concentrations of Hyflo DE at 30psig filtration pressure

This relationship was also observed and confirmed during the bentonite clay testing. At suspended solids content of 0.25wt% bentonite clay, the maximum filtration flux rate was measured when the body feed concentration was equal to 0.25 wt.% (Figure 8).

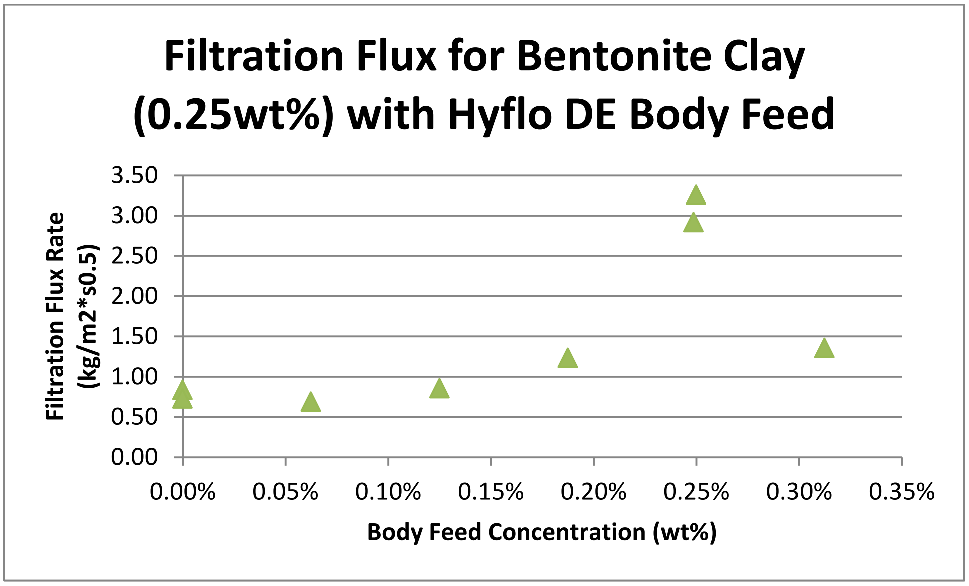

Figure 8: Average filtration flux rate for bentonite clay (0.25 wt.%) with various concentrations of Hyflo DE at 30psig filtration pressure

CONCLUSIONS

Based on the case study results, the optimal starting point of the body feed to suspended solids ratio is recommended to be 1:1 (g filter aid : g suspended solids). This optimal mixture was proven to be true in both the corn starch slurry and the bentonite clay slurries at various suspended solids concentrations.

While this equal parts concentration is optimal for the filtration rate, feeding less filter aid may be more beneficial for reducing filter aid consumption and filtration operating expenses. In the case of the 1wt% corn starch testing, having a ratio of 1:2 (g of body feed: g of suspended solids) resulted in a filtration rate reduction of only 4%. For the 0.5wt% solids test, the same body feed ratio resulted in only a 7% reduction in the filtration rate. By reducing the body feed ratio, there is only a slight compromise to filtration throughput, but a 50% reduction in body feed consumption. Evaluation of the impacts of reducing body feed should be evaluated on a case by case basis to determine the operating cost reduction versus the capital expenditure gains for a larger filter operating at a filtration rate below the optimal point. Reducing the body feed ratio will also decrease the solids load to the filter. In turn this can potentially increase the total filtration time during the filtration cycle; it will take longer to reach the maximum filter cake height. A longer filtration cycle can help improve the run time utilization of the candle filter or plate filter.

New and existing processes looking to introduce body feed into the filtration process should perform similar testing using the BHS Pocket Leaf Filter on a benchtop scale. Performing filtration tests such as these requires little time but can provide significant capital and/or operating cost savings. While this study was limited mainly to filter aid concentration there are many other variables that can be evaluated to optimize the filtration process: filter media, filtration pressure, slurry temperature, filter aid type and grade, use of precoat, precoat thickness, body feed concentration, filtration time/final cake thickness, and many more. All of these variables can be systematically tested using a well thought out test plan.

Results from these benchtop scale tests can easily be translated to a full scale BHS candle filter or BHS pressure plate filter to meet any clarification process needs. These automated technologies provide a significant advantage compared to other pressure filter technologies in the market. Through the use of automation, the BHS filters are able to be used efficiently with no wasted time which leads to a maximum throughput per unit area. With each filtration system built to order, the filters and packages are fully customizable to each process needs.

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