Continuous Vacuum Filtration Technologies for Sulfuric and Hydrochloric Acid Slurries

Presented at the 2016 AICHE ANNUAL MEETING ID# 452395

Garrett Bergquist, Process & Application Engineer
BHS-Sonthofen Inc., Charlotte, NC
E-mail: garrett.bergquist@bhs-filtration.com

Introduction

In many chemical manufacturing processes there exist slurry streams where the separation of solids from liquids is required. The range of equipment available for these streams is vast and the decision between equipment choices is not always easy. For clarification of low solids streams there exists technology such as candle filters, plate filters, and cartridge filters. These are typically a batch operation but can be made continuous by using multiple units in parallel. While one unit is actively filtering, the second unit is on side time operation such as filling, draining, or discharge. For higher solids applications continuous filters can be employed, including centrifuges, rotary vacuum filters, and horizontal belt filters.

Regardless of the specified filter type, when designing the filter, consideration must be made in the selection of appropriate materials. Materials of construction should be selected to withstand the temperature and chemical corrosion properties of the surrounding environment and liquids that come in contact with the filter.

This article discusses the use of alternate materials available in the construction of vacuum belt filters to address chemical compatibility concerns in applications using sulfuric acid and hydrochloric acid as the liquid constituent in a slurry. This article continues with a case study where the challenge is to process 2,200 kg/h dry solids that have been precipitated from a sulfuric acid solution.

Sulfuric Acid

Sulfuric acid is commonly and widely used in the production of fertilizer. Other manufacturing processes employ sulfuric acid to produce automotive batteries, pigments, iron, paper pulp, etc. Sulfuric acid can be a tricky liquid when it comes to designing robust systems for chemical compatibility. At high concentrations, sulfuric acid acts as a strong oxidizing agent. At low concentrations sulfuric acid acts as a reducing agent. For that reason, a material that is suitable for sulfuric acid at a high concentration may not be suitable for sulfuric acid at a low concentration and vice versa. Even at temperatures below 140^{0}F (60^{0}C), sulfuric acid at concentrations between 30wt% and 75wt% will corrode 316 stainless steel at rates greater than 200 mils per year (Figure 1).

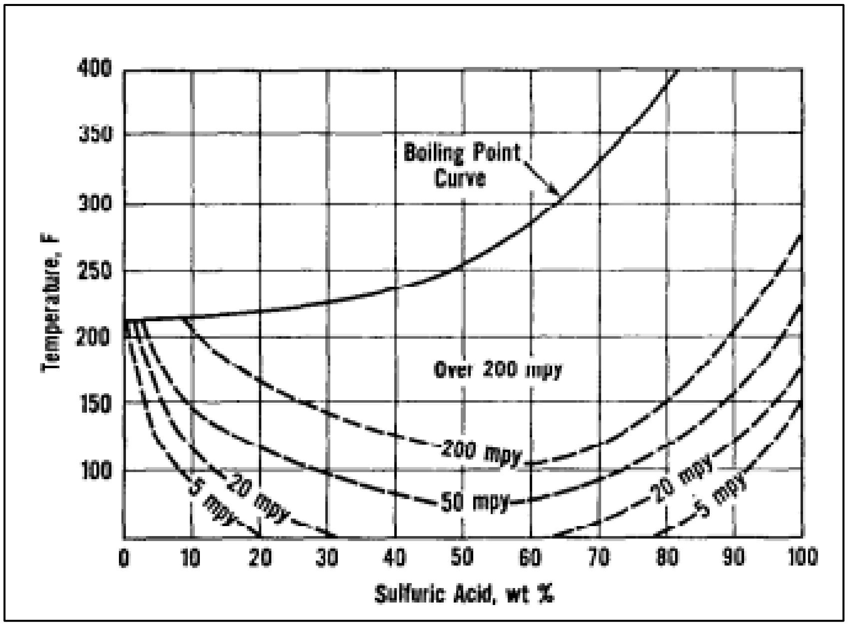

Figure 1. Corrosion of 316 Stainless Steel by sulfuric acid as a function of temperature. (Fontana, 1952)

When it comes to safety, sulfuric acid is an irritant and is considered very hazardous. In cases of skin contact and eye contact, burns may occur. Inhalation can result in severe irritation of the respiratory tract. Engineering controls are recommended to prevent physical interaction and to keep airborne concentrations of vapors below 1 mg/m^3 (NIOSH, 1997).

Hydrochloric Acid

Hydrochloric acid, also known as muriatic acid, is widely used in steel pickling, food production, calcium chloride production, gold mining, catalyst regeneration, dyes and pigment production, etc. Like sulfuric acid, hydrochloric acid is extremely corrosive to metals, including carbon steel, stainless steel, nickel and aluminum. In processes using hydrochloric acid, piping is often lined in thermoplastic and storage tanks rubber lined or made from FRP.

Hydrochloric acid is a highly hazardous substance and corrosive to mucus membranes. Exposure, even to vapors, can cause severe burns to exposed skin. The American Conference of Governmental Industrial Hygienists (ACGIH) recommends that the threshold limit value (TLV) for hydrochloric acid vapors to be 2 ppm (ACGIH, 2003).

Vacuum Filtration Technology

In all vacuum filtration technologies, the concept and mechanism of filtration is the same. Vacuum is pulled through a filter cloth backed by a porous supporting structure while slurry is fed to the cloth. The liquid filtrate is pulled through the cloth by the vacuum pressure differential while the solids are retained on the surface of the cloth. The gas and liquid that pass through the cloth are then piped together to a gas/liquid separator to allow the liquid to fall via gravity which is then pumped from the bottom of the receiver. The separated gas is piped from the top of the separator to the vacuum pump and subsequently exhausted (Figure 2). The configuration in which this process is accomplished is what sets apart the different vacuum filtration technologies.

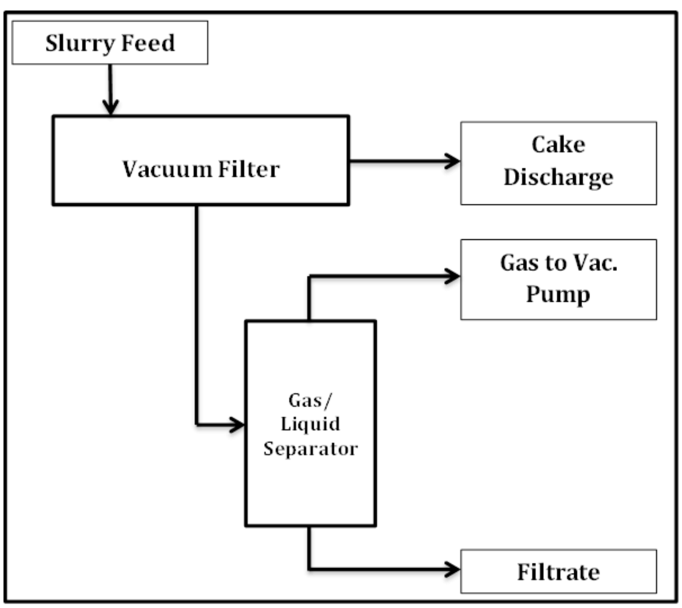

Figure 2. Process flow diagram for basic vacuum filtration

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Rotary Vacuum Drum Filter

In a rotary vacuum drum filter, filter cloth surrounds a cylindrical segmented drum. The drum is constructed from an alloy material and acts as the porous supporting structure for the filter cloth. The bottom part of the drum is submerged in an open slurry trough. With vacuum being pulled through the cloth, filtration begins to occur as the drum rotates into the slurry trough. Cake heights are determined by the residence time spent submerged in slurry. As the drum rotates the cake can undergo washing via spray nozzles and dewatering by pulling residual moisture from the cake via gas flow from the vacuum pressure differential. The cake is then discharged from the media by a scraper knife or discharge roller.

Benefits in using a vacuum drum filters include having a large amount of filter area in a small footprint relative to horizontal belt filters. Additionally with the discharge design, a cake heel can be left on the drum which allows for slight depth filtration at the beginning of the following filtration cycle. When it comes to operation using hazardous chemicals, specifically sulfuric acid and hydrochloric acid, the vacuum drum filter has some major shortcomings. For one, the slurry trough and acid rich filter cake are open to the surrounding environment. If the acid is at all heated above ambient temperature, then a significant quantity of acid vapor will fill the surrounding environment. Given the height of the filter, providing a ventilated enclosure to keep acid fumes below the respective exposure limits is both difficult and expensive.

A second drawback comes when considering compatible materials for the vacuum belt filter piping and drum. Stainless steel alloys such as 304 and 316 are not suitable for use in sulfuric and hydrochloric acid solutions. As previously stated, with relatively low temperature a broad range of sulfuric acid concentrations can corrode stainless steel over 200 mils per year. This corrosion consideration leads to the material of construction specification of higher grade alloys such as Alloy 20 or Hastelloy. By using more exotic alloys the cost of the filter is increased significantly. When comparing the cost of Alloy 20 and Hastelloy C-276, the relative cost is over 3 and 7 times that of 316 Stainless Steel respectively (Grocki, 2012).

Continuous Horizontal Rubber Belt Filter

In the continuous horizontal rubber belt filter, an endless filter cloth is backed by an endless, perforated rubber drainage belt which in turn is supported by a deck attached to the filter frame. The supporting deck is made of rollers or the rubber belt is floated by blowing air or slide water to reduce friction and wear as the rubber belt travels across it. The surface of the rubber belt is grooved to allow filtrate flow towards the belt perforations which are sealed to a vacuum box underneath the rubber belt. Motion of both the rubber belt and filter cloth are continuous at a constant speed.

The horizontal rubber belt filter is well utilized in fast settling slurries as the solids are fed onto the top of the belt vs the bottom feed mechanism of the vacuum drum filter. Additionally if intensive cake washing or longer drying times is required, the length of the belt filter can be designed to accommodate these process requirements.

When considering the use of the horizontal rubber belt filter for slurries with sulfuric and hydrochloric acid, some of the concerns regarding environmental exposure and material of construction can be addressed. A fume hood over the filter can be constructed more easily given the horizontal surface. However since the rubber belt must periodically be replaced via the side of the machine, the hood can only be suspended over the filter and not attached to the frame. Curtains can be hung from the edges of the fume hood to cover the remaining space but do not eliminate the risk of someone reaching in to the filter or overflow at the slurry feed zone in an upset condition. Additionally, to ensure material compatibility, different grades of rubber can be selected for the drainage belt material. However for sulfuric and hydrochloric acid, standard rubber materials such as natural rubber, SBR, and NBR are incompatible to various degrees. Like the vacuum drum filter, higher grades of materials must be considered for the rubber belt such as EPDM (Moss Rubber, 2005). Using these higher grade materials add significant cost to what is already most expensive piece of the rubber belt filter.

BHS Continuous-Indexing Vacuum Belt Filter

In the continuous-indexing vacuum belt filter, an endless filter cloth is backed by a stationary perforated filter bed. During operation the belt moves only periodically, step-wise down the filter bed. When the belt is stationary, vacuum is applied through the filter media to allow filtration. When the belt is indexing the vacuum is vented to allow motion across the stationary bed.

Like the rubber belt filter, the indexing belt filter is well utilized in fast settling slurries. Intensive washing can be performed like the rubber belt filter. There is a slight advantage for the indexing belt filter when it comes to countercurrent wash schemes as the wash additions and filtrates are better separated with the indexing design. Also with the indexing filter since the belt is stationary for a period of time, a press can be utilized to squeeze the solids while simultaneously blowing air through the cake to remove all remaining free moisture. The drawback to the indexing design is that side time introduced during the atmospheric pressure belt movement which reduces the filtration capacity compared to a continuous rubber belt filter design. For that reason the indexing belt filter it is better suited for medium to slower filtering slurries compared to ideal continuous rubber belt filter applications.

When considering the use of this design for hazardous slurries, the indexing belt filter is a perfect example of flexibility in both environmental considerations and available materials of construction. Because the filter bed is stationary and the cloth can be changed from the discharge end of the filter, a full hood is able to be built over the entire surface of the filter without side gaps (Image 1). This is a significant benefit when designing the filter for sulfuric or hydrochloric acid slurries as it completely isolates any interaction with the product. Through the use of blower a slight negative pressure can be maintained over the filter at all times. Alternatively the indexing design can also provide for a full gas tight enclosure which is beneficial for filtration when explosion proofing must be considered. The gas tight enclosure is able to be nitrogen purged and maintained using a nitrogen recirculation skid.

When considering materials of construction, the indexing design is able to be built from a multitude of polymers which commonly include polyethylene, polypropylene, or PVDF. These materials are able to be used for 100% of the wetted surfaces of the filter. The chemical resistance against sulfuric and hydrochloric acid with this selection of polymers is excellent at all concentrations (“Chemical Compatibility Database”, 2016). Additionally the material cost of these polymers compared to high grade alloys such as Alloy 20 and Hastelloy is much less. In fact, the use of polypropylene is even cheaper than construction using 316 stainless steel. The main limitation for using polymer constructed belt filters is temperature resistance. For a polypropylene belt filter, the maximum operating temperature is 175^0F – 194^0F (80^0C-90^0C) and 212^0F – 230^0F (100^0C-110^0C) for PVDF. However, for most vacuum filter applications this is not an issue as temperatures are often below the boiling point of water.

Image 1. BHS polypropylene indexing belt filter with oscillating slurry feed, fume hood and pressing/blowing device

BHS Case Study – Sulfuric Acid Filtration

A company producing solid chemicals used in paper coatings was using a process in which product dissolved in sulfuric acid (H_2SO_4) is precipitated using a neutralization step via NaOH. This company was looking to move away from NaOH neutralization as the price of NaOH had increased significantly over the past year. An alternative production method was known in which the concentration of sulfuric acid was diluted with water to approx. 25wt%. The dilution then allowed the product to precipitate from solution. With this method any residual sulfuric acid must be removed from the solids as all downstream equipment was constructed of stainless steel. Additionally the precipitated solids are fragile so no intense agitating or centrifugation was possible. Benchtop scale testing using a 20cm^2 pocket leaf filter (PLF) was performed to determine the appropriate technology, sizing, wash requirements and filter cloth to meet the application requirements.

PROCESS DATA

Slurry Throughput:	12,500 l/hr	(55 gpm)
Slurry Density:	1.3 g/mL	(30% H_2SO_4)
Suspended Solids:	13.5 wt%	
Dry Solids Throughput:	2,200 kg/h	
H_2SO_4 Concentration:	22-30 %	
Slurry Temperature:	max. 140ºF	(60^{0}C)

Through benchtop scale testing for the vacuum belt filter with the 20cm^2 PLF the following data was measured:

Filter Cloth:	80 µm polypropylene
Test Filter Area (A_f):	0.002m^2 (20cm^2)
Slurry Feed:	425 g
Filtration Time:	11 seconds
Mother Filtrate Quantity:	175 mL
Wash Volume:	235 mL
Wash Time:	55 seconds
Wash Filtrate:	238 mL
Drying time:	20 seconds
Heel Filtrate:	10mL
Cake Height:	50 mm
Wet Cake Weight:	185 g
Residual Cake Moisture:	65 wt%
Dry Cake Weight (m):	65 g

It was determined through preliminary calculation that a BF100 with 1.0 meter wide active belt width and an area of $A_{zone} = 0.75m^2$ per zone was appropriately sized. The cycle time and zone arrangement scale up for the BF100 at a filter cake height at 50mm was performed as follows:

Solids loading per stroke:

$$\frac{m}{A_F} * A_{Zone} = \frac{0.065kg}{0.002m^2} * 0.75\frac{m^2}{Stroke} = 24.38\frac{kg}{Stroke}$$

Number of strokes per hour:

$$N = \frac{2200kg/h}{24.38kg/Stroke} = 90Strokes/h$$

Duration of one cycle:

$$t_{Cycle} = \frac{3600\frac{s}{h}}{90\frac{Strokes}{h}} = 40\frac{\sec}{stroke}$$

With an indexing time of 5 sec with no applied vacuum, the active processing time per cycle is 35 sec. The belt filter zone arrangement based on an active time of 35 seconds is recommended as follows:

Table 1. BHS Continuous-Indexing Belt Filter BF100 Zone Arrangement

Process Steps	Test Times	Theoretical No. of Zones	Actual No. of Zones
Feed	-	-	1
Filtration	11 sec	0.31	1
Wash	55 sec	1.57	2
Drying	20 sec	0.57	1
Total	-	-	5 zones

With a total number of 5 zones required at $0.75m^2$ each, a belt filter BF100-038 with $3.75m^2$ in filter area was recommended to process 2,200 kg/h dry solids from the sulfuric acid solution (Image 2). A feed zone at the beginning of the filter with no vacuum was also recommended to allow for a more uniform solids distribution.

Image 2. BHS Polypropylene belt filter with fume hood built for the filtration, washing, and drying of the sulfuric acid slurry.

Conclusion

When considering new technology or a new production processes it is always important to verify that the specified materials match the chemical and temperature resistance requirements for each stream. While the technology in the market place for vacuum filtration is often well known, when it comes to dealing with hazardous slurries such as those containing sulfuric or hydrochloric acid the options should be carefully considered. Production areas containing acids should be fully enclosed to prevent splashing or spills in an upset condition, and to capture any hazardous acid vapors. Special attention must be made to specify compatible materials as stainless and carbon steels are often incompatible with most sulfuric and hydrochloric acid concentrations. However specifying compatible specialty alloys such as Alloy 20 and Hastelloy for wetted parts becomes very expensive quickly. With traditional filters such as the rotary drum filter or the continuous rubber belt filter providing containment and identifying economic compatible materials can be a near impossible task. With the continuous-indexing vacuum belt filter, fully enclosed or even gas tight operation is easily achievable. More importantly, through the use of polymer wetted parts such as polypropylene or PVDF the cost of the filter is able to be kept low, even lower than 316 stainless steel construction. Polymer constructed belt filters in hazardous acid environments have a proven track record of safe and reliable operation with low costs of purchase.

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