<u>Clarification Upsets and Ion Exchange</u> <u>Resin Symptoms Due to Design</u>

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Abstract:

The Ion Exchange Resin unit operation is an integral portion to any sugar refinery providing upwards of 80% of incoming liquor color removal. To achieve this color removal, it is imperative that proper flow rates are maintained, pressures do not exceed resin column thresholds, and regenerations are conducted successfully. Several refineries, despite having assorted designs, have had to overcome major obstacles to achieve these parameters. From mechanical and installation flaws plus lingering upstream contaminates remaining inside of the column, operating ion exchange resin columns can be time consuming and budget intensive. This paper will focus on key aspects of the column design which have contributed to these difficulties and how to manage them.

Summary

This paper will highlight the challenges that ion exchange resin (IER) decolorization columns will face because of upstream process upsets and certain design choices. Some of these challenges have been caused by excessive amounts of calcium carbonate carried over from press filtration to the IER columns. The columns experience either sudden impacts to performance which causes a premature shutdown or retention of the calcium carbonate which degrades resin performance over time. The refinery suffers in terms of overall melt performance due to pressure drop in the IER columns or reduced decolorization and higher washes in the pan house centrifuges. Fixing these problems results in significant maintenance expenditure, reduced refinery throughput and loss of resources as they work on corrective actions. To avoid these hardships, several measures have taken place at ASR refineries to either reduce or eliminate the losses that these problems can cause.

Introduction

The Baltimore plant is a 6.7 million lb/day melt capacity carbonatation refinery and processes sugar from North and South America. An integral part of this refinery, is ion exchange resin

decolorization, commonly referred to as decolorization columns. Commissioned in 2014, these 4 packed IER columns use a strong base anion resin which is necessary to remove color molecules from the incoming liquor. Each column contains 25 m³ of resin with a quaternary ammonium functional group designed to remove up to 80% of the incoming color from the wash house and clarification effluent. Since commissioning, several different challenges have arisen, due to upstream effluent components in combination with column design options. Two key design elements that affected performance were: filtration nozzles made of brittle fiberglass components that were subject to mechanical failure leading to resin loss, and a design of backwash outlet nozzles that limited backwash flowrates in the regeneration process, causing retained fines in the bed. These factors, exacerbated by calcium carbonate present in the influent liquor stream has been shown to lead to a reduction in color removal, lower throughput capacity due to the breakdown of resin beads which generates resin fines, and the accumulation of calcium carbonate conglomerates leading to throughput reduction. It is important to implement effective and sustainable solutions to these problems to maintain the best possible refinery performance. While solutions are important, having sound design applications goes a long way to preventing these long-term problems.

Reinforced Fiberglass Nozzles Initial Install

Each of the four Decolorization columns was fitted with more than 1,300 reinforced fiberglass nozzles with small slots to allow the liquor to pass through while keeping the resin beads inside of the column. Fiberglass nozzles were initially chosen for several reasons. The first reason was the cost comparison between fiberglass and stainless-steel nozzles. Also, fiberglass nozzles are more resistant to acid which offers benefits during in-situ acid treatments. These 1,300 nozzles were divided among three plates positioned at the bottom, middle and top of the column. As per the pictures below, the nozzles have some distinct shapes:

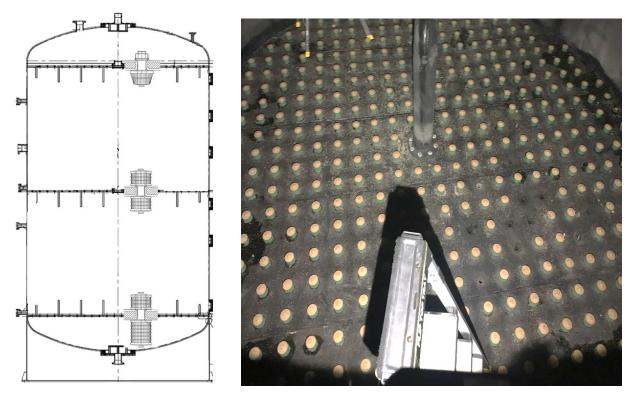


Figure 1: Column Drawing and Nozzle Install

The photo above shows several holes inside of the plate formerly occupied by the nozzles. Each one of these holes represents a nozzle that was either compromised or missing entirely. Also, the position of these nozzles provides its own challenges. Nozzles installed on the bottom of the columns were subjected of the calcium carbonate carry over from the clarification/filtration process. The nozzles at this position were susceptible to damage during both operation and column maintenance.



Figure 2: Examples of CaCO3 Build Up on Nozzles

When one single nozzle breaks it leads to a total IER column shutdown reducing the refinery throughout by approximately one-third of its total capacity. These nozzle breakages can also lead to resin beds with different ages mixing thus having aged resin inside of a fresh bed if a middle plate nozzle breaks.

Here at the Baltimore refinery, there were a total of 38 broken nozzles across 14 different events which led the graph below shows the nozzle breakages events over a 4-year span. These nozzle breakages led to approximately \$200,000 worth of labor costs and even more in lost production.



Figure 3: Interior of Nozzle with CaCO3 Build Up

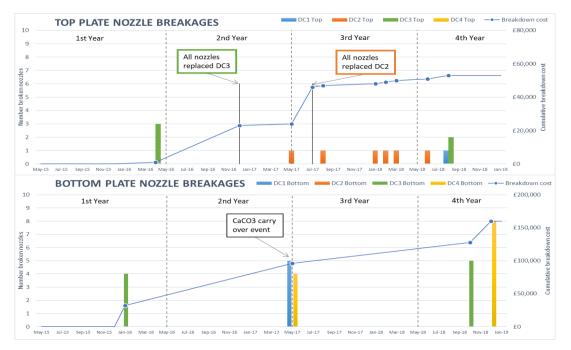


Figure 4: Data on Nozzle Breakages 2015-2019

Experts on the field advised that the service lifetime of fiberglass nozzles is 2.5 - 5 years depending on the process, and they need to be replaced as part of maintenance routine. When six or seven nozzles fail at once, it is time to replace the whole set. Given the frequency and challenges of even one nozzle failure, it was time to look for alternatives. The one viable alternative was replacing the fiberglass nozzles with a stainless-steel option which the site chose to do. The advantages and disadvantages of stainless steel nozzles will be discussed further in this paper.

Improvements and Drawbacks with Stainless Steel Nozzles

As a viable replacement for the fiberglass nozzles, all nozzles in the columns were replaced with stainless steel wedge wire nozzles. Without changing the nozzles, the estimated maintenance and downtime costs were projected to reach approximately \$1,000,000 over the course of 10 years. However, the nozzle upgrade was not cheap with the initial upfront cost of purchase and installation of \$480,000. There are several pros with the stainless-steel nozzles design. One is their increased mechanical durability in real-world process conditions. Since the installation of the stainless-steel nozzles in 2019, there have been no instances of a nozzle breakage. Another benefit is the time allotted to maintenance. Stainless Steel nozzles have shown to require less maintenance than their fiberglass counterparts. Additional refineries in ASR Group have had stainless steel nozzles applications for upwards of 10 years with no signs of damage or corrosion. Compared to a 2.3-5-year life span for the fiberglass nozzles the longevity proves to be a big advantage.



Figure 5: Stainless-Steel Nozzle Design – Top Nozzle (left), and Middle and Bottom Nozzle (right)

These stainless steel nozzles though more robust, provide several drawbacks. The nozzles on both the bottom plate and middle plate are composed of a dual wedge wire assembly comprised of 4 gaskets and just a single 3/8th inch all-thread to hold the assembly together. The installation of this assembly across the column proved to be sensitive to accurate installation, if the nozzles are not installed perfectly straight, it could lead to premature failure and resin loss.



Figure 6: Examples of Misaligned Nozzles

As can be seen from the above photographs, misaligned nozzles can result when the nozzles are not placed flush against the securing plate. These misaligned nozzles, depending on the severity, can lead to ion exchange resin leakage out of the column. As mentioned previously, resin leakage can cause premature shutdown of a column reducing throughput. To address this risk procedures have been updated to include a post nozzle install inspection procedure where team members inspect all the nozzles for alignment. Additionally, a torque setting was specified for each nozzle to avoid under or over torquing. Mechanical modifications were made to the top plate nozzles to address the situation where there were only two points of contact between the metal bracket, we call the 'T-Bar', and the gaskets. These two points of contact became compromised over a 3-year span which led to increased susceptibility to nozzle misalignment. To compensate for this a modification was made to the install to add a more even distribution of pressure throughout each nozzle. The pictures below show the progression of this top nozzle continuous improvement install.



Figure 7: Initial Top Nozzle (top left). T-Bar damage (top right). New Top Nozzle Design (bottom left) Top Nozzle Install (bottom right)

Calcium Carbonate Carryover

As mentioned previously with the nozzle install, one of the big challenges faced is how to deal with the presence of calcium carbonate from the clarification/filtration process in the Decolorization feed stream. Issues in the filtration step will lead the passing calcium carbonate causing blockages in the tight spaces in the nozzles and can be very difficult to eliminate. The pictures below show the level of buildup present on and inside of the stainless-steel nozzles.



Figure 8: Nozzle Buildup – External (left) and Internal (right)

This build-up came with a detrimental effect of a severely reduced throughput. As a result of this builds up the throughput from ion exchange was reduced at times to 25% of the target flow rate of the Decolorization column. Clearing out the blockages is challenging and requires a significant financial and personnel resources. While Baltimore team tried to clean the nozzles by dissolving the calcium carbonate with an in-situ acid treatment, resulting in some throughput improvement the most impactful action was the replacement of all the distribution nozzles resulting in multiple days of downtime.

Now that the issue has been corrected several different measures are ongoing to reduce the amount of calcium carbonate carryover from the filtration process. At the Baltimore facility there are plans to install an online turbidity meter. This will be used to quickly address any filtration upset and prevent build up in the IER column nozzles rather than allow the build-up to accumulate. At present turbidity measurements are completed several times per shift in the lab. Also, the clarification process is under tighter pH control with regular checking of the accuracy of the instruments on the carbonatation reactors to reduce the risk of generating small calcium carbonate flocs. Some of our sister ASR plants have multimedia filters upstream of the Decolorization process, which help prevent residual calcium carbonate from entering the columns. Currently the Baltimore refinery has a filter bag upstream of the columns which does help with capturing the passing calcium carbonate from filtration, but there is room for improvement in terms of the size of the bag filter. A smaller mesh will result in more calcium carbonate captured, but more frequent bag filters to be changed out.

The Criticality of an Effective Backwash

Arguably the most crucial step in regenerating the resin inside of the column, is performing an effective backwash. Proper backwashing is key to removing portions of the calcium carbonate carry over which build up in the resin over the service cycle. Other contaminates include resin fragments, or fines, which form over time. Failure to remove these suspended solids will cause

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flow restrictions due to resin bed channeling and middle and top plate nozzle blockages during service. The small particulates will make their way through the resin bed and get lodged into the nozzles thus causing the blockage. Key factors of the backwash are an optimum linear velocity across the bed section, high water flow rates and sufficient volume necessary to have an effective backwash.

There are different assorted designs when it comes to backwash hardware. Resin columns are commonly fitted with either a lateral network of many small discharge points distributed across the column (London and Toronto plants), or a single point large wedge wire strainer (Baltimore plant). The single point strainer causes a significant problem. During operation there is a stagnant area where the column contaminates are not filtered out. Over time, these contaminates continue to build up and can cause an accumulation of suspended solids resulting in increased pressure drop and reduced flow. The photos below show both the effective backwashing area with a single point strainer design along with the backwash lateral install.

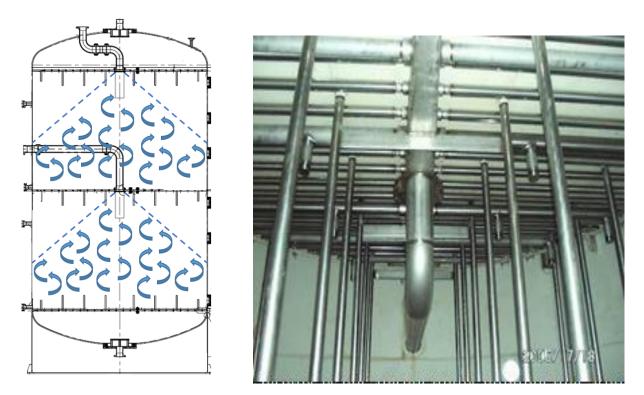


Figure 9: Backwash Area with a Single Point Strainer (left). Backwash Lateral Install (right)

The stagnant area of the single point strainer can contain resin fragments or additional calcium carbonate which does not get washed out during the backwash. Even with multiple backwashes, there are contaminates which may not be subjected to the backwash. The next photo shows the accumulation of calcium carbonate inside of a bed.



Figure 10: Evidence of Buildup

To manage the effectiveness of the backwash, the required flowrate and volume parameters need to be maintained. At the Baltimore facility there is an external tank which is used to acid treat the resin beds to remove the accumulation of calcium carbonate. Also, the external tank helps with the removal of additional particulates due to its higher freeboard the resin bed can be expanded more than in the column, but this, however, requires an extended regeneration time keeping the column out of service for longer. The best path forward is to move to a backwash application equipped with laterals. The backwash laterals design will cover most of the cross section of the top of the resin bed, thus minimizing the stagnant area resin bed

Selecting the Right Materials for the Application

As with any application, selecting the correct materials for the job is of the utmost importance. Your material should be able to withstand the stresses inside of the column in addition to not interfering with the filtration process. Common materials used inside of the resin column include both white and black nitrile materials specifically around internal column piping. While these materials are suitable for food applications, their ability to stretch can prove to be troublesome. The pressure in the column can exceed 50 psi which exerts a tremendous amount of force around areas in the column. The picture below shows just how the stresses of Ion Exchange resin column can affect the column components



Figure 11 Damaged Internal Gasket

Through incorrect installation, this gasket was damaged heavily which led to significant amounts of resin loss during the IER column backwash. To prevent resin loss, the backwash step was skipped while the repair was planned and executed, but that took several weeks to execute. As a result of that suspended solids were not removed during this time and there was a large amount of resin loss through the backwash piping.

When exploring new materials for the gaskets, Gylon gaskets were an effective alternative. Not only are they made of food grade material, but they are much more durable when it comes to the ion exchange resin application. Since installation, several months ago there is no evidence of a gasket failure during a backwash.

Ways Forward to Manage these Challenges

Refineries subjected to these challenges are best suited to find resources to address the root causes quickly. By monitoring the clarification process, any upsets which will cause downstream problems can be addressed at source, benefitting all downstream processes in the refinery. Basic parameters to keep into control are pH, lime ratio and incoming carbon dioxide content in the saturators, and final turbidity after filtration. The Baltimore refinery has plans to install a post filtration turbidity meter to manage the risk of calcium carbonate slip.

It is important to perform regular acid treatments of the resin beds. A low concentration HCl solution washed over the resin is very effective in removing calcium carbonate present in the resin bed, as well as other contaminants. The Baltimore facility not only acid treats the beds but will occasionally acid treat the IER columns to dissolve as much calcium carbonate as possible.

While substituting new materials for more robust materials is an effective way to make improvements, it is also important to have a robust preventative maintenance (PM) program of the ion exchange resin columns. Regular checks should be performed on a yearly basis to inspect filtration hardware, backwashing piping looking for evidence of blockages or mechanical damage. Expecting some internal column failures and getting ahead of the problem will only ensure the life of the asset and the day-to-day performance. IER column PM programs should include an inspection of the effluent system, total column cleaning during end-of-life resin changes, and routine replacements of any failing parts. While the PM procedure can be daunting, the work over several days of maintenance can save your refinery hundreds of thousands of dollars in unexpected maintenance cost and thus keep your asset in top shape.

<u>Summary</u>

In summary, there have been several different challenges when operating ion exchange resin columns at the Baltimore refinery. When designing ion exchange resin plants, it is of utmost importance to consider several key factors: a) correct selection of the internal column filtration hardware in the design stage, considering both mechanical strength, and materials of construction for the service challenges likely in practice b) designing for regenerating the resin

with an effective backwash, which will extend resin life and performance while managing pressure drop, in particular avoiding unbackwashed zones in the column with a single backwash point of exit, and c) implementing a robust PM program which includes not only thorough inspections but procedures to replace components that specify torque settings to reduce the risk of failure in what can be thousands of internal components. Though applying large amounts of resources can provide a temporary solution, selecting the correct design will help ion exchange resins perform to its maximum capacity.