

# RESTORING THE OPERATIONAL CAPACITY OF A COUPLED-LOOP MOLASSES DESUGARIZATION SEPARATOR

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## **Abstract:**

Maintaining extract product quality while maximizing recovery of the sucrose in the second (extract) loop was becoming increasingly difficult at the American Crystal Sugar Hillsboro molasses desugarization (MDS) facility. Molasses processing rates were reduced to accommodate increasing pressure drops across the second loop. Frequent high pressure shut-downs were leading to increased downtime and poorer separation in the second loop. Fractal plates were removed from second loop cells and viewed under a microscope. Scale was collected and analyzed. Scale was also found in the product stream feeding the second loop. Analysis of the scale indicated it was predominantly calcium oxalate. A fractal plate testing cart was built that allowed a comparison in performance between the scaled (fouled) fractal plates and new plates. Poor flow characteristics were observed in the fouled plates. Several approaches were tried to clean up the fractal plates with varying degrees of success. When the fouled plates were cleaned mechanically, better flow characteristics were observed. The process of mechanical cleaning was utilized in the factory. Pressure drops were markedly lower (as much as 60% less) after clean-up. Work is underway to determine the root cause of the scale that appears from time to time and fouls the separator. This paper reviews the troubleshooting and resolution of a problem that has plagued the process over time.

## **Introduction:**

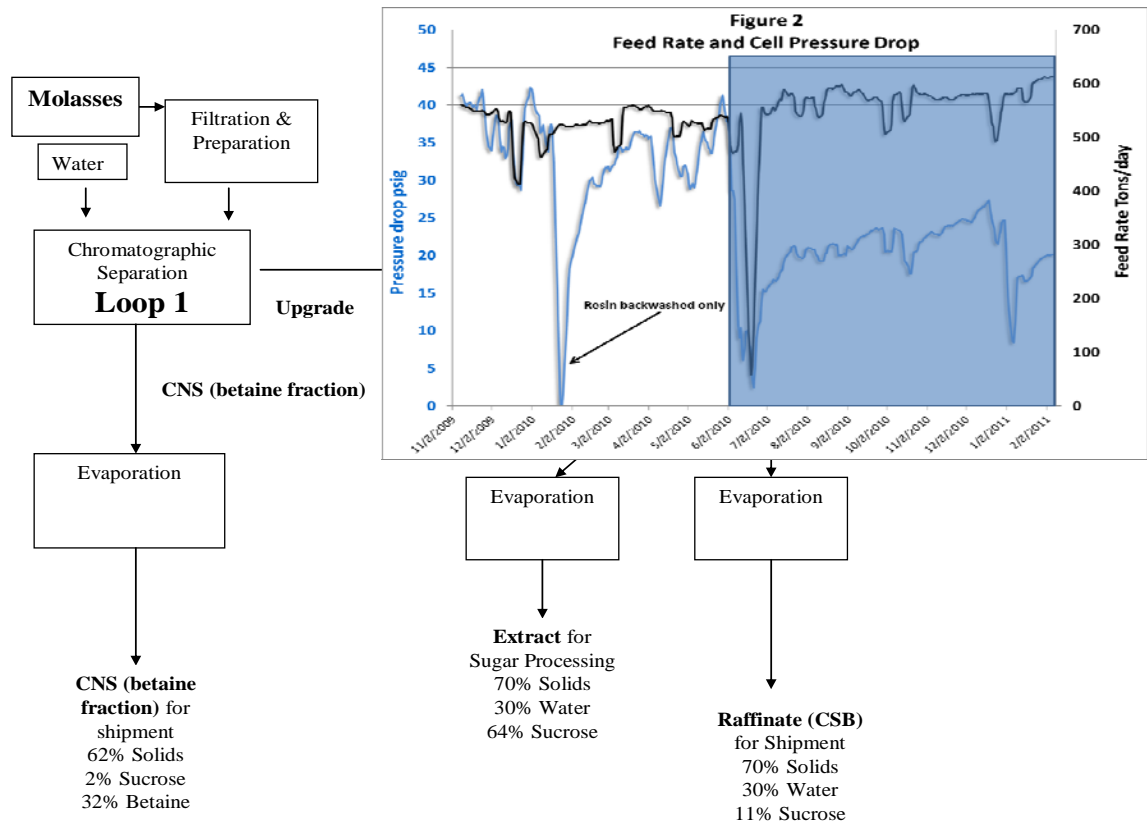
American Crystal Sugar Company (ACSC) operates an Amalgamated Research (ARi) coupled loop molasses desugarization (MDS) facility at Hillsboro, North Dakota. The Hillsboro separator was brought on line in January of 2000 and attained expected performance by July of 2000. The coupled-loop technology utilizes displacement chromatography along with simulated moving bed (SMB) technology (Kearney, 1997). Coupled loop is also known as multi-component simulated moving bed; the technology consists of two equilibrium curves established in the first loop. One curve consists of betaine and other amino acids also known as crossover nonsugars (CNS). This product is typically sold into the animal feed market. The other curve called the upgrade fraction contains the salts and sucrose. The upgrade material is processed in the second loop where sucrose is separated from the salts. The sucrose rich extract fraction is either stored or fed directly to the sugar end. The salt rich fraction known as raffinate or concentrated separator by-product (CSB) is concentrated and sold into the animal feed market.

As mentioned earlier, there are two distinct operations involved in the coupled loop or multi-component SMB molasses desugarization separator. What leaves the first loop impacts the second loop. Conversely, since we do not store upgrade, the second loop can impact the first loop with regard to processing throughput.

Figure 1 below provides a basic overview of the coupled-loop separator at Hillsboro. Soft molasses is fed into the first loop. Leaving the first loop are the CNS (betaine-rich) and Upgrade

(sucrose-rich) fractions. The Upgrade goes into the second loop where it is separated into the sucrose-rich Extract fraction and the Raffinate fraction. White sugar is crystallized from the Extract fraction. Raffinate (CSB) is concentrated and sold as a by-product. The CNS from loop one is also concentrated and sold into the animal feed market.

**Figure 1**



The MDS plant at Hillsboro has had a reliable operating record; in the past few years we have been operating the plant 360 days a year. Over time we have noticed an increase in operating pressures at a given molasses feed rate. To accommodate increased operating pressures, the processing rate eventually was decreased. More frequent shutdowns were experienced due to high pressures. Eventually, up to 12 percent of rated operating capacity was lost.

**Results and Discussion:**

In response to increased pressure drops observed in the extract loop, a more aggressive resin backwashing schedule was used for loop two. Resin backwashing helped initially, but elevated pressure drops quickly returned; the impact of resin backwashing was short-lived. Subsequently, resin traps continued to require frequent changes; a short shutdown occurs every

time a trap is changed which is not desirable. Ultimately, the ability to optimize control parameters was affected. As an example, step time could not be adjusted properly in order to accommodate higher pressure. Figure 2 on the right shows the pressure drop trend in blue (psig) and the processing rate per day in tons of standard molasses in black. Pressure drop data are from one cell in loop two but is representative of what was observed in all cells. On the left side of the graph, note the rather rapid rise in pressure drop after resin backwashing. Eight weeks after backwashing it was typical to observe the pressure drop increase from 20 psi to 40 psi. With increased pressure drop, overall system pressure was elevated. Typically train operating pressures moved from 40 psi to 70 plus psi resulting in temporary shutdowns. The area of the graph in blue background will be addressed later.

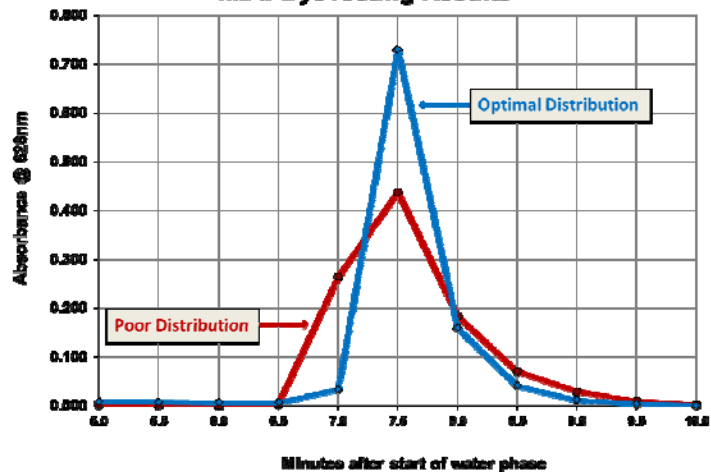
**Resin and Distributor Assessment:**

A thorough review was done on factors that impact operating pressure of an MDS train. One of the ways we check the system for resin and distributor integrity is dye testing in conjunction with pressure drop monitoring and resin trap change outs. Dye testing provides insight into internal fluid distribution in a separator cell. We used a modified dye test method introduced to ACSC by Amalgamated Research. A solution of Blue dye #1 was injected into a cell during the water phase. A series of samples were collected at the end of one cell and read in a spectrophotometer at 628nm. Since it was difficult to introduce the exact amount of dye injected into each cell tested, the absorbance data were “normalized” then plotted so that peak area total was the same for each cell tested. Sharper absorbance peaks indicate better fluid distribution resulting in improved component separation and better overall performance. Broader peaks indicate poor internal flow characteristics resulting in a “smearing” of components; hence, poorer separation results. Figure 3 illustrates the results from dye testing done on a cell with poor (red) distribution and another with optimal distribution (blue). The sharp peak (blue line) is expected with clean, properly packed resin and a sound distribution system. Poor fluid distribution can be due to dirty, broken, or unpacked resin, fouled internal distribution components, broken internal components, poor control, faulty pumps, partially plugged resin traps and leaking valves.

Items such as molasses quality, internal distribution system of the cells, condition of the resin in the cells, and flow control, among other issues, were also considered. Since operating pressure problems were present only in the extract loop, we focused our efforts there. The items reviewed and results obtained were as follows:

1. Resin in the cells must be uniformly distributed. There should be a minimum of broken beads or resin fines present. Results: We found that the cells were uniformly packed and a minimum of resin fines were present. Dye testing results were poorer than expected after backwashing resin.

**Figure 3  
MDS DyeTesting Results**



2. Mechanical components such as hoses, distributors, fractal plates, and valves must be in good repair and operating properly. Flow meters should be in calibration; pump and valve control loops must be properly tuned. Results: Hoses and valves were not contributing to pressure drop. Fractal plates appeared to have surface fouling. The pressure differential across the fractal distributors was higher than expected.
3. Loading of the separator must be appropriate for the volume of resin in the system; as the resin is overloaded, separation performance decreases. Results: Throughput was less than design capacity; resin levels in cells were adequate.
4. Operating temperatures must be in the correct range to avoid microbial infections and ensure proper kinetics. Results: Temperatures were at target levels. No sign of bacterial infection was present.
5. Pretreatment of the feed molasses must meet minimum standards. Suspended solids must not be introduced into the system; liquids entering the cells must be degassed. Result: No deficiencies were found in molasses feed or liquids entering trains.

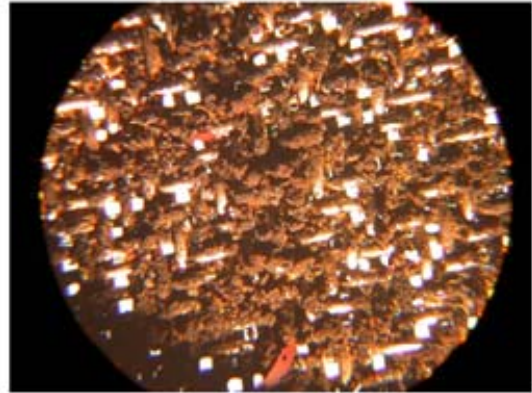
Our investigation ruled out most of the aforementioned with the exception of internal distribution component fouling. With elevated pressure drops across the resin bed, frequent resin trap changes were required. Resin was removed from a cell that had been operating with a high differential pressure. While the resin was being backwashed, the internal components of the cell were scrutinized. Upon inspection of the fractal screens with a hand held microscope, it was apparent some of the screens were plugged. Fractal plates were removed from the cells for further inspection. Samples of the scale and solids found in resin traps and fractal plates were analyzed. The material found was predominantly whewellite ( $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ ) a form of calcium oxalate. The samples were analyzed by a local university (NDSU) using X-Ray Powder Diffraction (XRPD) to determine the types of crystalline phases present. Interestingly, the scale was present only in the upper internal components of the cells. Two fractal plates are shown in the photo to the upper right. The fractal plate on the left is from the upper distribution system in a cell from the extract loop. Note the dark coloration of the plate. The plate on the right is from the lower distribution system in the same cell. Very little discoloration has occurred. In addition, there was virtually no scale found in that fractal. The color of the bottom fractal looks like a new fractal. Various treatments for cleaning up the upper fractals were tried.

### **Remedies:**

Some fractals were soaked in a mild acid (2% HCl) solution; others were soaked in a mild (2%) caustic solution. The most effective cleaning was realized with the caustic solution. The photo below-right shows the material that was flushed out of one upper fractal plate after it

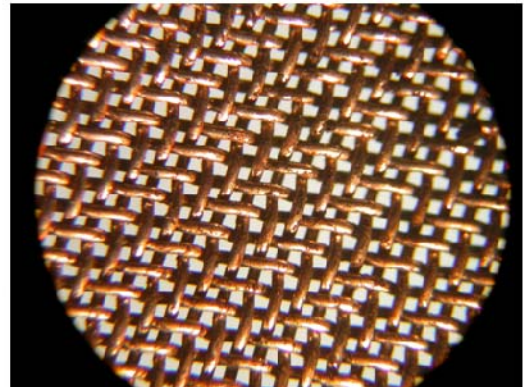


had been soaked in a 2% caustic solution (room temperature) for 1 hour. While it appeared that the upper fractals were fouled, it was unknown to what degree the fouling impacted performance of the fractal plates. In order to assess the plates, a testing apparatus (test cart) was built. The test cart pumped water through a fractal plate; flow rate and pressure were monitored. Data were collected on pressure drop across a fractal plate at a given flow rate. With fractal technology turbulent flow was not expected. We monitored fluctuations in pressure, which are an indication of turbulent flow. New plates, upper plates that had been in service and lower plates that had been in service were tested. Pressure was monitored at flow rates that ranged from 3 to 8.5 gallons per minute.



Results from fractal testing:

- There were no pressure fluctuations and flow from all ports was very uniform for new fractals.
- Upper fractals that had been in service exhibited turbulent flow patterns and increased pressure drop as flow rates increased.
- Lower fractals that had been in service compared favorably to new fractals.



Fouled upper fractal plates were cut open to view internal fouling. Distributor screens were removed to view the inside of the screens and the scale in the ports. Port diameter was reduced up to 15% in the upper fractals that had signs of fouling. Scale and debris on the screens was considerable. The photo to the right shows the interior of two screens that had been removed from an upper fractal plate. Roughly half the open area of each screen was blocked with debris. Port diameter (at the screen) of a new fractal is 0.096 inch; screen size is typically 0.875 inch.



Photomicrographs were also taken of screens removed. To the right is a photomicrograph (30X) of the inside of a screen removed from an upper fractal. Backlighting was used to identify open area in the screen. The majority of what should be open area is covered with scale and debris that is consistent with the appearance of calcium oxalate.

While chemical treatment with caustic seemed to loosen up scale and debris, it was not a first choice in exporting a method to the factory. Several mechanical approaches to cleaning up

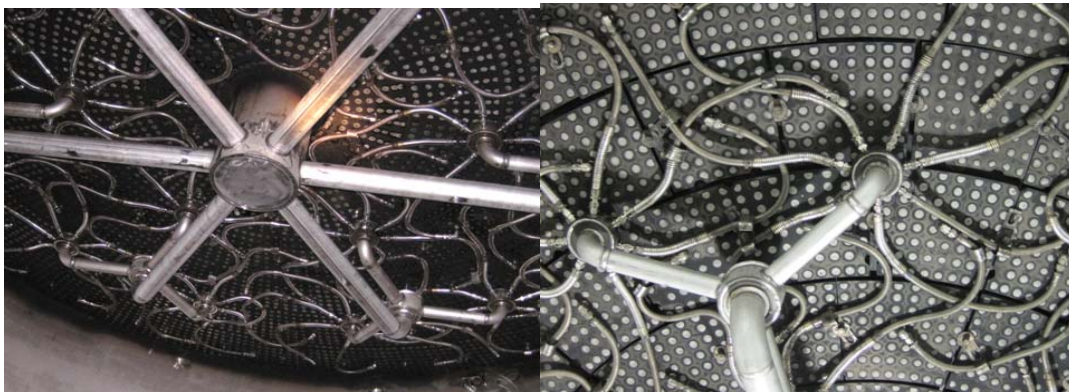
the fractals were considered. Pressure washing was tested on upper fractal plates that had been removed. Different spray distances were tested to gage the impact of pressure washing on the fractal screens. When using room temperature water at 1200 psi and a fan spray pattern nozzle at 10 to 12 inches from the fractals, no screen damage occurred.

After pressure washing, the fractals were back-flushed to loosen and flush out debris. Finally, the cleaned up fractals were assessed on the test cart. Fractals that had been fouled and performed poorly on the test cart before pressure washing performed much better afterward. To the right is a photomicrograph of a screen that had been pressure washed. There is still evidence of scale on the screen but the open area has been restored.

Based on the results obtained in the pilot plant setting, a method for cleaning up the fractals was exported to the Hillsboro factory site. First, a cell in the second loop was isolated and the resin removed to a backwash tank. After the cell was cooled and ventilated, the upper fractals were pressure washed in place with 1200 psi water. A nozzle was used that provided a fan spray pattern.

After the upper fractal plates were cleaned, the cell was closed up. Hot soft water was run into the cell at a rate of 1000 gallons per minute in an up-flow mode; normal flow through the upper fractals is in the down-flow configuration. Back-flushing of the cell continued for 30 minutes. During the back-flushing routine it was not unusual to see floor drain area covered with debris that had been removed from the distribution system. During distributor clean-up, resin was also thoroughly backwashed.

Below are photos of the upper distribution headers before (left) and after (right) pressure washing. In the photo of the cell before pressure washing notice the brownish-black material present on the fractal plates, hoses, and distribution pipes. Anywhere from two to four hours were required to pressure wash the upper distribution system.



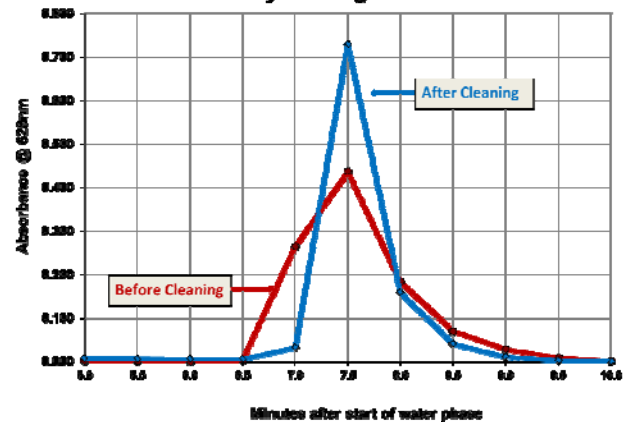
## Results from Pressure Washing and Back-flushing Cells:

Pressure drops decreased dramatically in each cell that was pressure-washed while the resin was backwashed. Typically the average pressure drop across the cell dropped from 40 psi to 10 psi. The addition of pressure washing and back-flushing to the maintenance routine decreased pressure drops by as much as 60 percent. High operating pressure shutdowns that had become routine became non-existent and remain so today. Since operating pressures decreased, step time could be shortened up to match the nonsugar load coming into the facility. Better sucrose recovery was realized while throughput was increased by 12 percent. The facility now operates above rated capacity (600 tons per day) by several tons per day. Downtime due to pressure shut-downs is now nonexistent; shutdowns for resin trap servicing have also been reduced. In Figure 4 to the right, processing rate of standard molasses (black line) is shown along with pressure drop across a cell (blue line). On the left side of the graph, note the rather rapid increase in pressure drop after backwashing the resin only. In contrast, on the right side of the graph, note that the increase in pressure drop over time is much slower when the fractals are pressure-washed while resin is backwashed. Before pressure washing was implemented, the pressure drop across a typical cell increased from 20 psi to 40 psi in about 8 weeks. When a cell's fractals were pressure-washed while resin was backwashed, initial pressure drop was lower. Average pressure drop increase (12 to 24 psi) was over 11 months of operation versus the 20 psi increase observed in 2 months prior to the new maintenance routine. Also note the increase in processing rate that was realized when fractal cleaning was done.

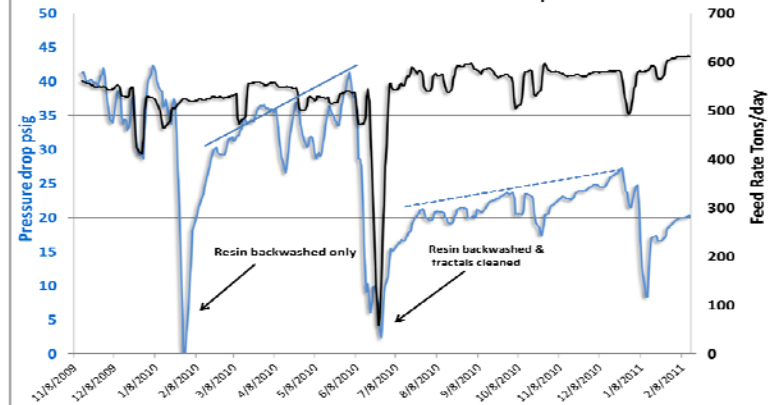
Follow up dye testing was completed on several cells in the second loop. Earlier in this paper a graph (Figure 3) representing results of dye testing illustrated the difference between poor fluid distribution and good distribution. The graph shown on the right (Figure 5) was based on data

collected from a cell before and after the upper fractals were pressure-washed and the resin backwashed; the same data displayed in Figure 3 earlier. This reinforces the improvement in internal fluid distribution realized when fractals were pressure washed before backwashed resin was introduced into the cell.

**Figure 5**  
**MDS Dye Testing Results**



**Figure 4**  
**Feed Rate and Cell Pressure Drop**



## **Conclusions:**

- The scaling/fouling issue was found predominantly in the second loop.
  - The precipitate was found in the concentrated upgrade check filters.
  - Precipitate was found in the upper fractals and in the resin traps in the second loop
- Calcium oxalate was identified as the principle component in the scale and precipitate.
- Calcium oxalate scale build-up was causing decreased performance of the MDS facility.
  - Processing rate was significantly reduced due to elevated operating pressures.
  - To accommodate elevated pressures, tuning control changes were made that negatively impacted component separation.
- Careful pressure-washing of the upper fractals and back-flushing the cells reduced differential pressure across resin beds and reduced overall operating pressure.
- Backwashing the resin was done concurrently with fractal cleaning to provide maximum benefit.
- By pressure-washing the fractals, back-flushing cells, and backwashing resin concurrently, rated operating capacity has been restored.
- Lower operating pressures allow for more flexibility in optimizing control parameters allowing for improved separation performance in second loop.

## **Acknowledgements:**

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## **References:**

1. *Kearney, M. (1997): Coupled loop chromatography. 29. General Meeting American Society of Sugar Beet Technologists.*