Introduction

A new continuous countercurrent ion exchanger was installed at the Sri Chamundeswari Sugars factory near Bangalore in India for the removal of color from sugar melt.

The raw sugar melt has a color of 400 ICUMSA with a Brix value of 64% and a density of 1273 kg/m³ at 75°C. The objective of this new ion exchanger was to reduce the color of the sugar melt with minimum 70% and reduce simultaneously the rinse water and chemical consumption. The available floor space was limited and the resin inventory had to be reduced.

PuriTech, a Belgian based company specialized in Continuous Countercurrent Ion eXchange better known as CCIX with its patented ION-IX system provided the solution for this client.

Existing technologies includes fixed bed, standard continuous and counterflow ion exchange systems are all based on a batch style operation. Fixed bed systems are used already for decades and applied in around 90% of the ion exchange projects, but compared with continuous countercurrent ion exchange systems their performance is relatively poor.

With reference to a case study at the Sri Chamundeswari Sugars factory, this paper provides a comparative analysis of CCIX with fixed bed systems.

Mechanical design of the ion exchanger

PuriTech’s in India’s based partner – Chemical Systems from New-Delhi - has built the ion exchange columns and piping with local manufacturers and subcontractors while PuriTech provided the process design, mechanical detailed design and the local assistance for the commissioning and process fine tuning.

During the fabrication of the stainless steel resin vessels, several changes were implemented to create an equal distributed flow through the ion exchange columns and to reduce or eliminate dead zones inside the resin vessels. These dead zones will seriously influence the performance of the continuous countercurrent ion exchanger because the rinse and regeneration flows are much lower in comparison with standard fixed bed system.

The spray nozzles inside the resin vessels were equally distributed over the bottom and top plates of the resin vessel.

The raw sugar melt was pre-filtered by 3 bag filters with a slotsize of 25 µm and a capacity of 16 m³/h each for a pressure drop of maximum 0.5 bar before sending it to the ion exchanger.

The 24 stainless steel resin vessels were installed in 4 rows of each 8 vessels symmetrically adjacent of the ION-IX multi port valve. Each resin vessel contained approx. 265 liters of color removal resin.

The ION-IX multi port valve

At the heart of the installation, the ION-IX multi port valve (Figure 1) is installed which distributes the sugar melt to 16 ion exchange vessels in parallel for adsorption of color but at the same time several resin vessels are in regeneration or in a rinse phase. During a full rotation of the process disk inside the ION-IX valve, each resin column is subjected to an entire sorption and desorption cycle.

The raw sugar melt flow enters the ION-IX valve at the top and leaves the ION-IX valve cleaned from most of the color from the outlets at the bottom of the ION-IX valve. At the side nozzles of the multi port valve, the resin columns are connected which are filled with resin (Figure 2).

The ION-IX valve was designed for a maximum operating temperature of 75°C with a pressure of up to 5 bars (60 psi).

Figure 1. Multi-port valve

Figure 2. 30 m³/h stainless steel continuous countercurrent ion exchanger
The process:

The basis for using ion exchange resins for colorants coming from cane juices is because most of the wide variety of colorants show a very favorable selectivity for strong base anion (SBA) resins. In addition, the removal of these color components from the resin can be accomplished fairly easily with a salt solution (typically 10% NaCl).

There are two types of SBA resins that can be used – Acrylic and Styrenic – type of resin. Acrylic resins are more resistant to fouling and are usually considered for the “dirtier” juices and typically achieve a 60 – 70% color removal. Styrenic resins will remove a slightly higher percentage of the color 70 – 80%, but Styrenic resins are less resistant to long term fouling and are usually used for the “cleaner” raw melt.

For this color removal project, the Styrenic Lanxess resin S 6368 A was selected and a total volume of 6325 liters have been used for a the treatment flow of 30 m³/h of sugar melt.

This Lanxess resin was of the Styrenic type because this type of resin has a higher decolorisation capacity in comparison with the Acrylic resin S 5428. The Styrenic resin type is more sensitive to fouling by organic compounds and require occasional acid regeneration. The decolorisation capacity of Styrenic resin is higher than that of Acrylic resin, but the color is not so efficiently removed during regeneration and the capacity to capture color can drop very rapidly if overloaded.

In the ION-IX system, the ION-IX multiport valve continuously distributes the sugar melt flow; the sweeten Off; the regenerant and the rinse streams through the stationary resin columns so that all the resin are in an active duty. The process “moves” in a circle, moving through each operation, one step at a time from one column to the next. The process rotational speed is proportional to the feed flow and the color removal requirement.

The ION-IX system for decolorisation of cane sugar can be broken up into 6 distinct process zones with each zone operating continuously. See Figure 3 of process on the HMI screen.

Process phase 1: Adsorption or loading zone

Hot sugar melt is fed to the ION-IX system under flow control and diverted to a number of ion exchange columns in parallel and in an up-flow direction. At Sri Chamundi, 16 columns were used. Since the sugar flow rates and viscosities are significantly higher than all the other process streams, the majority of the system is allocated to the adsorption zone. For sugar applications, up-flow of the sugar flow is preferred in order to minimize bed packing and high pressure drops due to the high viscosity of the sugar. For this project, a 1 pass parallel adsorption zone was applied. If higher color removal is required, a double pass adsorption in parallel will be applied.

Process phase 2: Adsorption wash or “Sweeten-Off”

After the resin has been loaded with color, the resin column moves towards the next process phase which is the adsorption wash also called “Sweeten-Off” phase. In this phase, the sugar which is still located inside the resin column is displaced from the resin column by using hot demineralized water while the color components remain loaded on the resin and does not come off in the sweet water effluent. This Sweeten-Off phase typically contains 2 columns configured in a series arrangement. The sweet water effluent can be recycled to the feed or upstream processing or combined with the produced sugar.

Process phase 3: Back wash & cross regeneration (included in the adsorption wash)

After adsorption wash, the resin column can be switched into a single column configuration where the resin can be backwashed and / or intermittently cross-regenerated with a diluted acid 4 – 5% HCL solution. Backwashing is necessary on an intermittent basis to keep the resin beds loose and to also purge debris and resin fines outside the resin bed. Cross-regeneration is necessary on an intermittent basis to reduce organic and inorganic resin fouling that is known to occur over time especially on the styrenic resin types.

Process phase 4: Regeneration

The SBA resin are regenerated with a hot salt solution in the regeneration zone. The salt solution is sodium chloride (8-10% NaCl) with a small amount of caustic (1% NaOH). The mechanism for color desorption is not stoichiometric, but is concentration dependent. The regeneration zone is configured as down flow, opposite of adsorption, to achieve maximum loading & stripping efficiency. The minimum regeneration time is about 0.5 hour. The columns in the regeneration zone are configured in series thereby maximizing the counter current contact between the regenerant and the resin.

After a resin column moves into the regeneration, the initial effluent liquid is clean water and can be easily recovered and reused in the process. The later regeneration effluent is a highly colored salt solution which should go to waste or to the Brine recovery system.

Process phase 5: Rinsing

After the resin has been regenerated, the resin column will move into the rinse zone. In this operation, the caustic brine is displaced from the resin column using clean hot demineralized water (condensate). The hot condensate water is fed to the ION-IX system for decolorisation of cane sugar can be broken up into 6 distinct process zones with each zone operating continuously. See Figure 3 of process on the HMI screen.
IX system under flow control. The rinse zone contains 3 columns configured in a series configuration. The rinse effluent is also sent to the Brine Recovery System, thus maximizing the usage of the recovered salt.

Process phase 6: Sweeten-On

After rinsing out the caustic brine, sugar is used to displace the clean water from the rinse phase inside the resin bed. This step helps reducing the sugar product dilution. After the resin is moved into the sweeten-On position, the initial effluent liquid is clean water that can be easily recovered and reused. The remaining sweeten-on effluent is a low brix sweet water and can also be recycled to upstream processing.

Process optimization of ion exchanger

During the process fine tuning, PuriTech’s process engineers performed several rinse and regeneration profiling. This profiling allows the process engineer to verify if the ion exchange vessel is over or under regenerated. The same profiling is done for the rinse phase. This process fine tuning allows to optimize the whole process and by doing that, chemical consumption and rinse waters are reduced as maximum as possible.

By sampling the outlet of the rinse and regeneration phase on a equally spread time, the composition of the rinse (or regenerant fluid) becomes clear. As shown in the Figure 4, the ink black fluid which was after an index of the ION-IX valve was transferred from the regeneration phase into the rinse phase is washed out of the ion exchanger. (samples from left to right)

After 6 samples, the fluid becomes more clear and the rinse water becomes visible in the transparent cups. At the end of the sampling, the outlet of the rinse phase is almost completely clear. The demineralised water (condensate for our project) used for the rinsing has pushed out the effluent (Brine + caustic + color waste from the sugar) from the ion exchanger.

Cross regeneration of Styrenic resin:

As the Styrenic resin has a higher tendency for organic fouling and color from sugar is mainly an organic component, Styrenic resin requires a periodic cross regeneration with an acid solution. A 4–5% HCl solution was used during cross regeneration. Each ion exchanger vessel was cross regenerated on a weekly basis.

Initially the color from the NaCl / NaOH regeneration breaks already through after 12–15 minutes (Figure 5) while the index time was 30 minutes which indicated that over cross regeneration was applied to the resin.

This breakthrough of regeneration fluid is expected at the last minutes of the profiling.

During cross regeneration with HCl, the extent of color removal from the resin is much higher and the duration is longer than during normal regeneration.

The high color profile in the last rinse phase is more concentrated and lasts for a longer period until the end of the index time. Possible crossover of color and regeneration fluid towards the sweeten-on zone could happen during the cross regeneration cycle.

It is advisable to increase the rinse flow during cross regeneration just to avoid cross-over of color or chemicals into the sugar flow

After the acid cleaning cycle, a normal regeneration cycle but with twice the standard NaOH concentration was done this to neutralize the remaining acid and to remove the acid adsorbed by the weak base sites of the resin.

Process performance data:

The continuous countercurrent ion exchanger has been

<table>
<thead>
<tr>
<th>Table 1. Comparative analysis of CCIX and Fixed Bed systems</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
<tr>
<td>Sugar melt flow</td>
</tr>
<tr>
<td>Color loading</td>
</tr>
<tr>
<td>N° of IX vessels</td>
</tr>
<tr>
<td>Switch next IX vessel</td>
</tr>
<tr>
<td>Resin volume</td>
</tr>
<tr>
<td>Rinse water</td>
</tr>
<tr>
<td>Regeneration</td>
</tr>
<tr>
<td>Sweeten off</td>
</tr>
<tr>
<td>Sweeten on</td>
</tr>
</tbody>
</table>
Operational for several months and it outperformed the standard fixed bed systems. The color fed to the system was higher. A comparison overview is given in Table 1:

With continuous systems, 1 BV (IX Resin Bed volume) of Sweeten Off flow is transferred towards the Sweeten ON phase to recover the bed volume of sugar during the next phase of the process. By doing this, the sugar does not become diluted with the rinse water column that has moved towards the adsorption zone.

The rinse water is re-used to dilute the concentrated brine and caustic to bring it to the correct concentration, so also this water is recovered in the system.

With the Brine recovery system installed, 80% of the brine is also recovered and sent back to the system. New salt + caustic is added to get the required concentration again so it can be used in the continuous ion exchanger. A salt and caustic reduction of more than 30% is achieved together with a resin inventory reduction of 60% and a less water consumption of more than 50% compared to a fixed bed system of the same size.

The sugar color reduction:

Sugar color during processing is reduced during different process steps. One of the last treatment phases is usually ion exchange.

Figure 6 shows the color reduction obtained just before clarification (left), the color and also the purity just before the ion exchanger (middle) and the color level in the sugar melt just after the new installed ion exchanger (right).

Conclusions

The new color removal installation at Shri Chamundeswari Sugars has been proven to meet its design criteria of low-waste production; salt and caustic usage; very low power consumption and also low operator intervention.

The 70% color reduction was initially reached when the production started with fresh, new resin but after several months the performance of the resin reduced and a color removal of 60% was still achieved.

Continuous Countercurrent Ion exchange is a huge improvement for the removal of color from sugar solution in comparison with standard fixed bed systems. The process performance is much better than regular fixed bed systems and reduces not only the capital cost by using smaller IX columns with less resin but also the operational cost substantially for the customer.

There remains potential within the system for further optimization. Following the initial operating period where confidence in the system was established, further optimization of the consumables and thus improving the OPEX even further is still feasible. The confidence has grown sufficiently with the customer as to the reliability of the ION-IX system.

Acknowledgements

A special thank to Mr. Anup Keserwani and R.K. Moharana from M/S Chemical Systems Technologies I Pvt Ltd, New Delhi who has supplied the complete system along with Melt Clarification and Multi Bed Filter and Brine Recovery System for the success of this project.

We thank also Mr S Srinivasan , MD of Sri Chamundeswari Sugars Ltd who has given us the chance to put our Continuous Counter Current ION-IX System in his factory which is the first in India and also to his technical team at the factory.